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EXPERIENCE IN THE SEPARATION AND PURIFICATION OF TRANSPLUTONIUM
ELEMENTS IN THE TRANSURANIUM PROCESSING PLANT AT THE
OAK RIDGE NATIONAL LABORATORY*

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The Transuranium Processing Plant (TRU)(1,2,3) at Oak Ridge National Laboratory (ORNL) is the production, storage and distribution center for the heavy-element research program of the U. S. Department of Energy (DOE) and its predecessors, the U. S. Energy Research and Development Administration, and the U. S. Atomic Energy Commission. TRU and the neighboring High Flux Isotope Reactor (HFIR) were built to produce quantities of the transuranium elements for use in research. Operations in both facilities were begun in 1966. Since then, TRU has been the main center of production for transcurium elements in the United States, producing 460 mg of ^{249}Bk , 4 g of ^{252}Cf , 18 mg of ^{253}Es , and 10 μg of ^{257}Fm . Target rods containing plutonium, americium, and curium are remotely fabricated at TRU, irradiated in the HFIR, and then processed at TRU for the separation and purification of the heavy actinide elements. All elements from plutonium through fermium are separated and purified. Portions of the plutonium, americium, and curium are refabricated into targets for additional irradiation. The berkelium,

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californium, einsteinium, and fermium are distributed to researchers. More than 1000 shipments of these materials have been made to about 30 different laboratories in the United States and several foreign countries. The complete production history of key isotopes is given in Table I. During 14 years of operation, 39 chemical processing campaigns have been completed at TRU to process about 265 targets which had been irradiated in the HFIR and about 195 targets which had been irradiated in a reactor at the Savannah River Plant (SRP). There are three distinct periods in the history of TRU operations. From 1966 to 1970, TRU had been recovering transuranium elements as rapidly as they could be produced in the HFIR and fabricating targets as fast as feed material was being recovered for recycle.

Production rates were limited because the feed materials that were available (^{242}Pu and curium containing predominantly ^{244}Cm) require long irradiation periods (up to 18 months) to produce appreciable amounts of the heavier elements. In the period from 1970 through 1973, in addition to the processing of irradiated HFIR targets, operations at TRU were expanded to include the processing of special SRP targets that had been irradiated as part of the Californium-I campaign, an irradiation and processing campaign to provide ^{252}Cf for use in an SRP program to evaluate the commercial market for ^{252}Cf . Approximately 720 mg of ^{252}Cf was recovered for the SRP program and 94 mg of ^{249}Bk and 5 ug of ^{254}Es recovered from the SRP targets was used in the

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50 research programs. Then, beginning in 1974, TRU operations reverted to the processing of targets fabricated at TRU and irradiated in the HFIR. However, since then, the rates of production of the transcurium isotopes have been considerably higher than in earlier years because some of the curium recovered from the Californium-I material has been used as feed for the HFIR targets. Berkelium, californium, einsteinium, and fermium are produced much more rapidly from this curium because it is rich in the heavier isotopes (curium-246-248) which can be transmuted to transcurium isotopes in shorter irradiation periods.

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TABLE I. KEY ISOTOPE PRODUCTION HISTORY AT TRU

Fiscal Year	²⁴² Pu (g)	²⁴³ Am (g)	²⁴⁴ Cm (g)	²⁴⁹ Bk (mg)	²⁵² Cf (mg)	²⁵³ Es (mg)	²⁵⁷ Fm (pg)
1967	87	25	134	0.34	5.6	0.014	0
1968	0	188	212	0.05	0.5	0	0
1969	15	5	57	2.2	15	0.1	0.07
1970	8	13	72	7.6	52	0.4	0.19
1971	10	3	439	37	284	0.7	0.72
1972	16	3	350	66	513	0.9	0.85
1973	5	4	240	49	428	1.6	1.25
1974	0	2	87	39	386	2.2	1.5
1975	0	3	104	75	717	3.8	1.6
1976	0	2	50	29	277	1.7	0.54
1977	0	0	38	52	499	2.6	1.1
1978	0	0.6	48	67	632	3.6	1.6
1979	0	3	21	32	322	0.8	0.6
Totals	141	252	1852	456	4131	18.4	10

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The heart of TRU is a battery of nine heavily shielded hot cells housed in a two-story building. Of the nine cells, four contain chemical processing equipment for dissolution, solvent extraction, ion exchange, and precipitation operations. Three contain equipment for the preparation and inspection of HFIR targets, and two cells are used for analytical chemistry operations. In addition, there are eight laboratories (four on each floor) used for process development, for process-control analyses, and for final product purification and packaging operations. Figure 1 shows a cross section of a typical cell and the surrounding building areas. The top and back of the cell line is enclosed by a high-bay area (the "limited access area") that is equipped with a bridge crane. Removable top plugs provide access to the cells. Service lines enter the cells through removable plugs in the back walls and tops. Shielded pits in the floor behind the cells house off-gas filters and a pipe tunnel for process lines. The front face of the cell line makes up one wall of the operating area. Essentially all process and building service instrumentation is located in the operating area. The second floor immediately over the operating area is a chemical makeup area for process-reagent head tanks, uncontaminated pumps, etc. Transmitters for process and service instrumentation are located in this area.

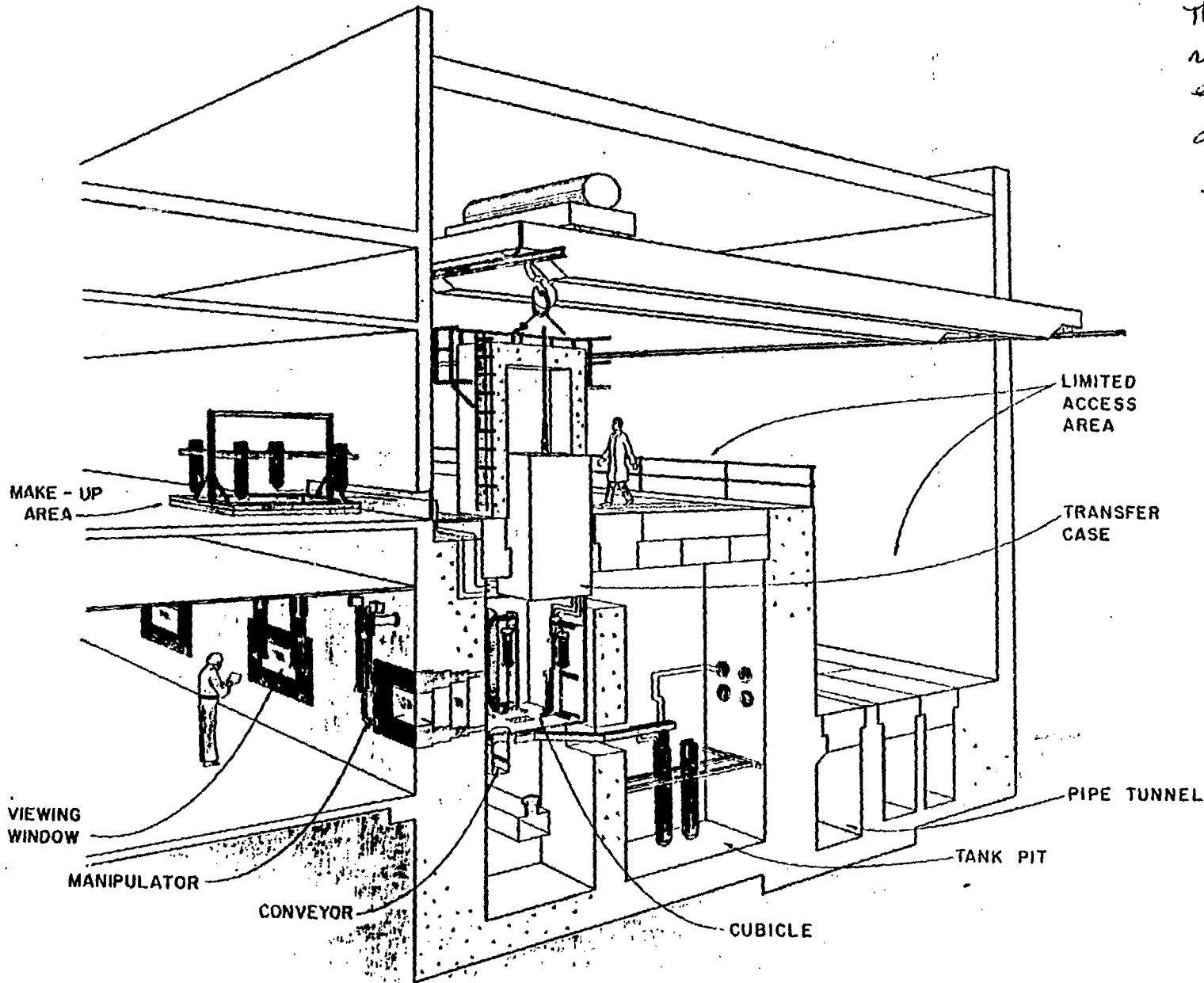
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~~FIG. 1. TRANSURANIUM PROCESSING PLANT~~

Fig. 1. Cross Section through a typical cell at TRU.

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Within each shielded cell, process equipment is enclosed in a fixed containment box (the cell cubicle) which is about a two meter cube. Small items of chemical processing equipment, such as valves, pumps, ion exchange columns, and solution sampling devices are mounted on racks in the cubicles. This equipment can be installed or removed remotely by using manipulators and air-operated impact wrenches. Contaminated equipment can be introduced into or removed from the cell cubicles through an intercell conveyor to a glove box or to a shielded carrier at a loading station at one end of the cell bank. Alternatively, equipment may be introduced or removed through the top of the cubicle by use of a shielded caisson (transfer case) designed to maintain shielding and contamination control during the transfer.

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A tank pit for housing waste collection equipment and process and storage tanks is located behind and below each cell cubicle and shielded from it by a concrete wall. The equipment in the tank pits is serviced and maintained by means of a combination of contact, remote, and underwater maintenance techniques.

The extensive provisions at TRU for changing and modifying equipment have allowed continual updating of the plant to include new concepts in chemical processes (4) and equipment design (5). Since the beginning of operation, the processing equipment in the cubicles has effectively been changed twice

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through replacement of 17 equipment racks in 9 cell positions. The flexibility and reliability of the chemical processing equipment and techniques have been improved significantly through this evolutionary process (6).

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Transuranium Element Chemical Processing

Since operation of TRU began, numerous process changes have been made to counteract problems caused by equipment corrosion, to satisfy new processing requirements, and to utilize improved processes. In initial operations at TRU, unexpected corrosion of the Zircaloy-2 equipment occurred in high specific-activity chloride solutions, and the corrosion-produced zirconium caused operating problems that made some of the flowsheets for the processing steps either difficult to operate or totally unusable. Because of these and other problems, several new flowsheets were devised or adopted, tested, and scaled to plant-size equipment.

Chemical processing to recover transuranium elements is accomplished in a series of separate steps which is called a processing campaign. In a typical campaign, about ten irradiated HFIR targets plus rework material from previous campaigns are processed over a period lasting about two months. The composition of the feed material is shown in Table II. Of the transplutonium elements in the feed, approximately 25% of the curium and 5% of the berkelium and californium are rework material from the previous campaign. In addition to the components shown, gram quantities of zirconium, nickel, iron, and

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chromium are frequently added from equipment corrosion and must be removed.

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TABLE II.
APPROXIMATE COMPOSITION OF TYPICAL TRU CAMPAIGN FEED:
TEN HFIR TARGETS PLUS REWORK MATERIAL

Component	Source	Weight (g)
Al	Target cladding, spacers, pellet matrix	1200
Cm ^a	Target residual, plus rework	65
Si	Activation of aluminum	25
Mo, Ru, Pd, Cs, Ba	Fission products	25 ^b
Rare-Earth Elements	Fission products	15 ^b
Ni	Impurity in aluminum	10
Zn	Impurity in aluminum	7
Fe	Impurity in aluminum	7
Mn, Cu, Mg, Cr, Ti	Impurity in aluminum	5 ^b

^aOther transplutonium elements in the feed include 1 g of Am, 30 mg of Bk, 300 mg of Cf, 1 mg of Es, and 1 pg of Fm.
^bTotal amount from the group.

The sequence of steps that is now being used successfully to process HFIR targets in the mainline cell bank is shown in Fig. 2. The processing steps generally tend to separate the transplutonium elements, as a group, from fission products and gross impurities before partitioning and purifying the elements. Wastes from the processing are transferred to the ORNL Intermediate Level Waste system for subsequent treatment and disposal.

Target Dissolution. A HFIR target is a 9.4-mm-diam by 0.89-m-long rod which is encased within a cylindrical tube which

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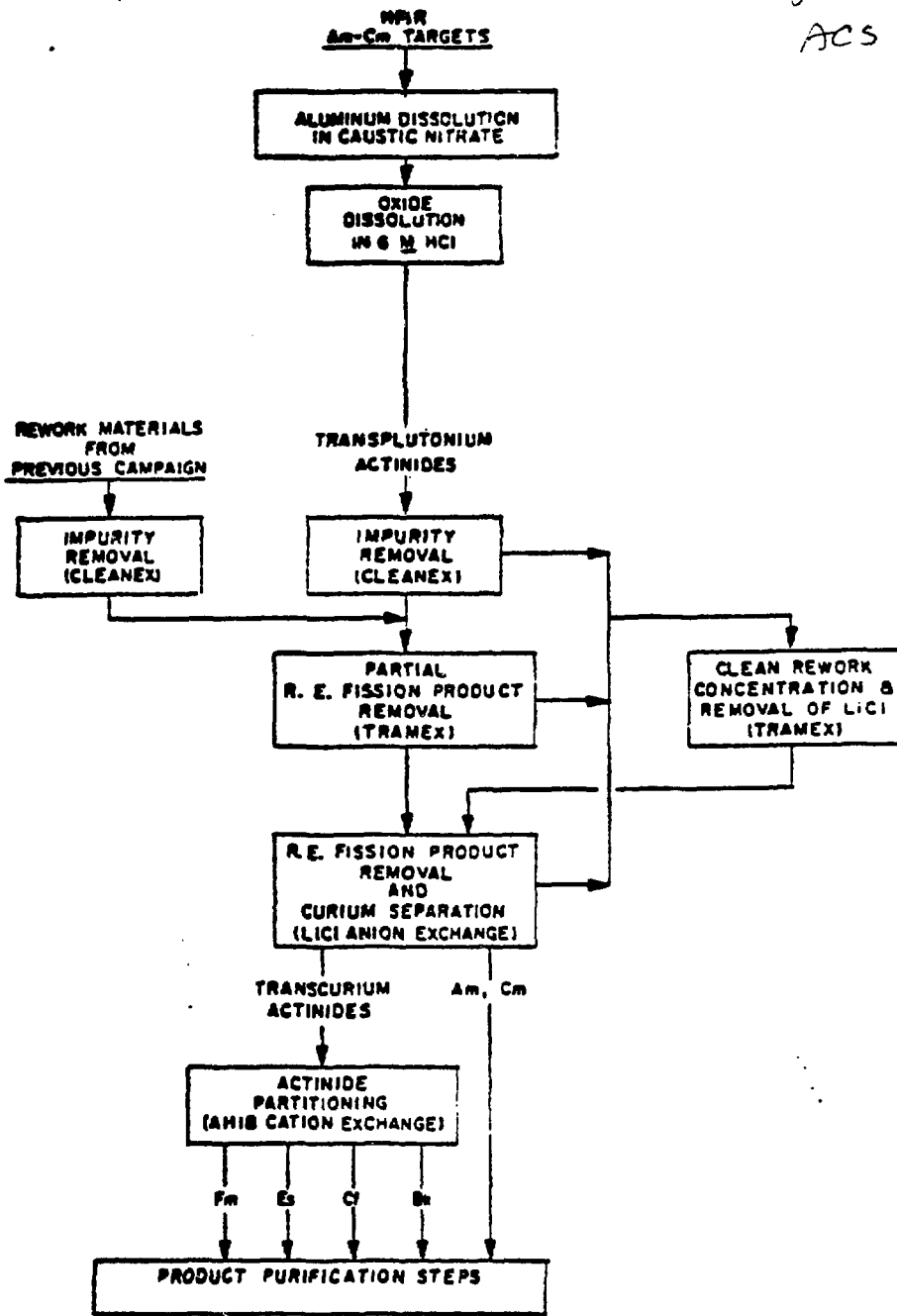


Fig. 2. Sequence of mainline steps used to process HFIR targets.

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serves to channel the flow of cooling water in the reactor. The coolant-flow tubes are mechanically removed from irradiated target rods before dissolution because the rate of dissolution of tubes was found to be more difficult to control than that of rods.

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A well-known, two-part method (7) is used for dissolution of targets. The aluminum is preferentially dissolved in caustic-nitrate solution and after the aluminum-bearing solution is removed, the residual oxides (transuranium elements and fission products) are dissolved in concentrated HNO_3 . The group of target rods is put into a water-jacketed Zircaloy-2 tank and covered with a solution of 2.1 M NaNO_3 (using a NaNO_3 -to-aluminum mole ratio of 1.2), which is then heated and held at 90 to 94°C while 10 M NaOH is added. The reaction is highly exothermic and the rate of dissolution, as indicated by the temperature difference between the solution in the dissolver tank and the water jacket, is controlled within empirically determined limits by varying the rate of addition of 10 M NaOH . The average dissolution rate is about 10 mol of aluminum per hour. Addition of NaOH is continued until a ratio of 2.4 mol of NaOH per mole of aluminum is reached. Approximately 25 liters of NaNO_3 and 10.8 liters of NaOH are required for 10 targets. Calculations made from laboratory data indicate that for each mole of aluminum dissolved, 0.37 mol of NH_3 and 0.02 mol of H_2 are evolved into the dissolver off-gas. These gases are

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diluted to nonexplosive concentrations by an air purge of the dissolver tank. The aluminum-bearing caustic-nitrate solution is pumped from the dissolver to a holding tank through a porous stainless steel filter. About 0.1% of the actinides are lost to the caustic-nitrate solution.

Concentrated HNO₃ and water are added to the dissolver vessel to comprise about 7 liters of 5.5 M HNO₃ which is simmered at 103°C for two hours to dissolve the actinides.

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A major product of a processing campaign is ²⁵³Es, which has a half life of 20.5 d. Thus, it is important to process HFIR targets after only short decay. Because of this the targets usually contain about 7 TBq of ¹³¹I at the start of a campaign.

About one-half of the radioiodine in the targets remains in the caustic-nitrate solution which is stabilized with sodium thiosulfate and stored until the radioiodine decays. During the acid dissolutions, the dissolver off-gas is directed to the iodine sorption system in which the gas is contacted with a 10 M HNO₃--0.4 M Hg(NO₃)₂ solution in an absorption column containing six bubble cap trays. Then, the iodine is evolved from the dissolver solution by simmering, air sparging, and H₂O₂ additions until the solution contains less than 0.2 TBq of ¹³¹I. Iodine is removed from the off-gas by a factor of about 500. The mercuric nitrate solution is neutralized and stored until the radioiodine decays and is then disposed of to the

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Intermediate Level Waste system.

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50 Miscellaneous Impurity Removal. The dissolver solution is treated by means of a two-stage Cleanex (8) batch solvent extraction to remove miscellaneous metallic impurities. The nitric acid dissolver solution is concentrated to a small volume to remove excess acid, HCl is added, and the residue is digested to dissolve zirconium precipitates. The solution is then adjusted to about 0.25 M acidity with NaOH. An oxidant, 1.5 M LiOCl, is added to bring the concentration to 0.1 M LiOCl to oxidize any molybdenum in the feed to the extractable Mo(VI) form. The adjusted feed solution is then contacted with an organic solution of about 35 liters of 1 M bis-2-ethylhexyl hydrogen phosphate (HDEHP) in normal paraffin diluent to extract the transuranium elements and 5 M NaOH is added to adjust the aqueous solution to about 0.03 M acidity to enhance the extraction coefficient. The aqueous phase is transferred to another tank where a second cycle of extraction is used to recover residual transuranium elements. Five liters of 1.6 M Adogen (an 8-10 carbon tertiary amine) in diethylbenzene (DEB) diluent is added to hold iron in the organic phase during stripping and the transuranium elements are stripped from the organic extract using a 6 M HCl--0.5 M H₂O₂ solution.

Rare-Earth Removal and Separation of Curium from Heavier Elements. The Cleanex product solution is combined with rework material from the previous campaign and the composite solution is processed by means of a Tramex (9) batch solvent extraction to

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obtain a product containing about 98% of the transcurium elements, 90% of the curium, and 10% of the rare earths. The batch Tramex process consists of the following: (a) adjusting the composite solution to about 10 liters of 12 M LiCl--0.04 M HCl; (b) extracting all transplutonium and rare-earth elements with about 20 liters of 1 M Adogen in diisopropylbenzene (DIPB); (c) adding about 20 liters of a solution of 0.6 M Adogen--0.03 M HCl--0.05 M dibutylhydroquinone in DEB to dilute the organic phase composition to 0.8 M Adogen; (d) scrubbing out 10% of the curium and 90% of the rare earths with 10.6 M LiCl--0.02 M HCl; and (e) stripping the organic extract with 8 M HCl--0.1 M H₂O₂ to recover the transplutonium elements. The scrub raffinate is stored temporarily and processed later with other clean rework.

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In preparation for subsequent anion-exchange runs, the Tramex product solution is treated by a two-step clarification process to eliminate problems with solids formation and significantly reduce the amount of actinide elements diverted into rework solutions. In the first step, the Tramex product solution is washed with DEB to remove entrained Adogen which would be degraded to a tar in the subsequent boiling step and sorb a significant amount of the transplutonium elements. In the second step, treatment of the Tramex product solution includes: (a) adjustment to 12 M LiCl--1 M HCl; (b) filtration to remove insoluble materials such as aluminum, zirconium, and sodium; (c)

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50 dilution by flushing the filter; and (d) readjustment to about 5 liters of 12 M LiCl--0.1 M HCl to provide a clear feed solution for the anion-exchange runs.

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The clarified Traxex product solution is divided into two or three batches (<35 g of curium or <19 g of ^{244}Cm per batch) and processed by LiCl-based anion exchange, which is discussed in detail in another paper at this symposium (10), to obtain further decontamination from rare earths and to separate curium from the heavier elements. In each run, the transplutonium and rare-earth elements are sorbed on Dowex 1-X10 ion exchange resin from a 12 M LiCl solution, rare earths are eluted with 10 M LiCl, curium with 9 M LiCl, and the transcurium elements with 8 M HCl. About 5% of the curium is purposely eluted along with the transcurium elements to prevent losses of ^{249}Bk , which elutes immediately after the curium and is not distinguishable by the in-line instrumentation. The transcurium element fractions from each run are combined and processed in a second-cycle run, using new resin, to remove most of the excess curium.

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Transcurium Element Separation. The transcurium elements are separated by means of a high pressure ion-exchange process, which is described in another paper at this symposium (11). Feed for this process (0.25 M HNO_3) is prepared by precipitating the transcurium element product from the second-cycle anion-exchange run with LiOH, filtering to separate the precipitated trans-

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50 curium element hydroxides from the LiCl-bearing solution, and
dissolving the filtered precipitate in HNO₃. About half of the
feed solution (≤ 200 mg of ²⁵²Cf per batch) is processed in each
of two high-pressure ion-exchange runs. In each run, the
transcurium elements are loaded onto Dowex 50W-X8 resin in a
short "loading" column (200 mm long) and then chromatographi-
cally eluted through a longer (1.2 m) column. This technique
reduces radiation damage to the resin in the long column and
35 enables reuse in several runs. The resin in the short column is
replaced after each run. The fermium, einsteinium, and califor-
nium are eluted with 0.25 M ammonium alphahydroxyisobutyrate
(AHIB) at pH 4.2, the berkelium with 0.25 M AHIB (pH 4.6), and
the residual curium with 0.50 M AHIB (pH 4.8). Product yields
are usually greater than 90%, and excellent separations are
obtained. The berkelium is decontaminated from californium by a
factor of 500 and the einsteinium is decontaminated from cali-
fornium by a factor of 10³ to 10⁴.

Rework Processing. Immediately following a campaign, all
clean solutions containing significant amounts of the transplu-
tonium elements (usually about 10% of each element in the cam-
paign feed) are accumulated and reprocessed. "Clean" solutions
are those that have a known history, such as raffinates from
process steps, and solutions which are used to flush the equip-
ment internally following the campaign. These solutions do not

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50 contain a large amount of corrosion products or other potentially troublesome components.

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Also, "dirty" rework materials are obtained from the product purification steps, target fabrication, and equipment flushing. Since numerous temporary piping connections are made to accommodate multipurpose use of several tanks during a campaign, leaks and/or small spills can occur. Thus, the cell cubicle floors and external portions of the equipment are flushed thoroughly after each campaign. All of the rework materials obtained between campaigns are accumulated, processed by means of one or two cycles of the Cleanex batch extraction process to remove miscellaneous impurities, and recycled to the next campaign.

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Product Finishing. The sequences of steps used to purify each of the products are shown in Fig. 3. A three-step sequence is used to purify the americium-curium (referred to hereafter as "curium" since very little americium is present normally) and convert this material to the oxide form for use in HFIR targets. The curium product solutions from each of the first cycle LiCl-based anion-exchange runs are combined and processed by means of a Tramex batch extraction to remove most of the LiCl. After the Tramex product is converted to a nitrate medium by adding HNO₃ and evaporating HCl, the curium solution is divided into batches, each containing 25 g of curium or less, and each batch is purified from miscellaneous impurities (predominantly

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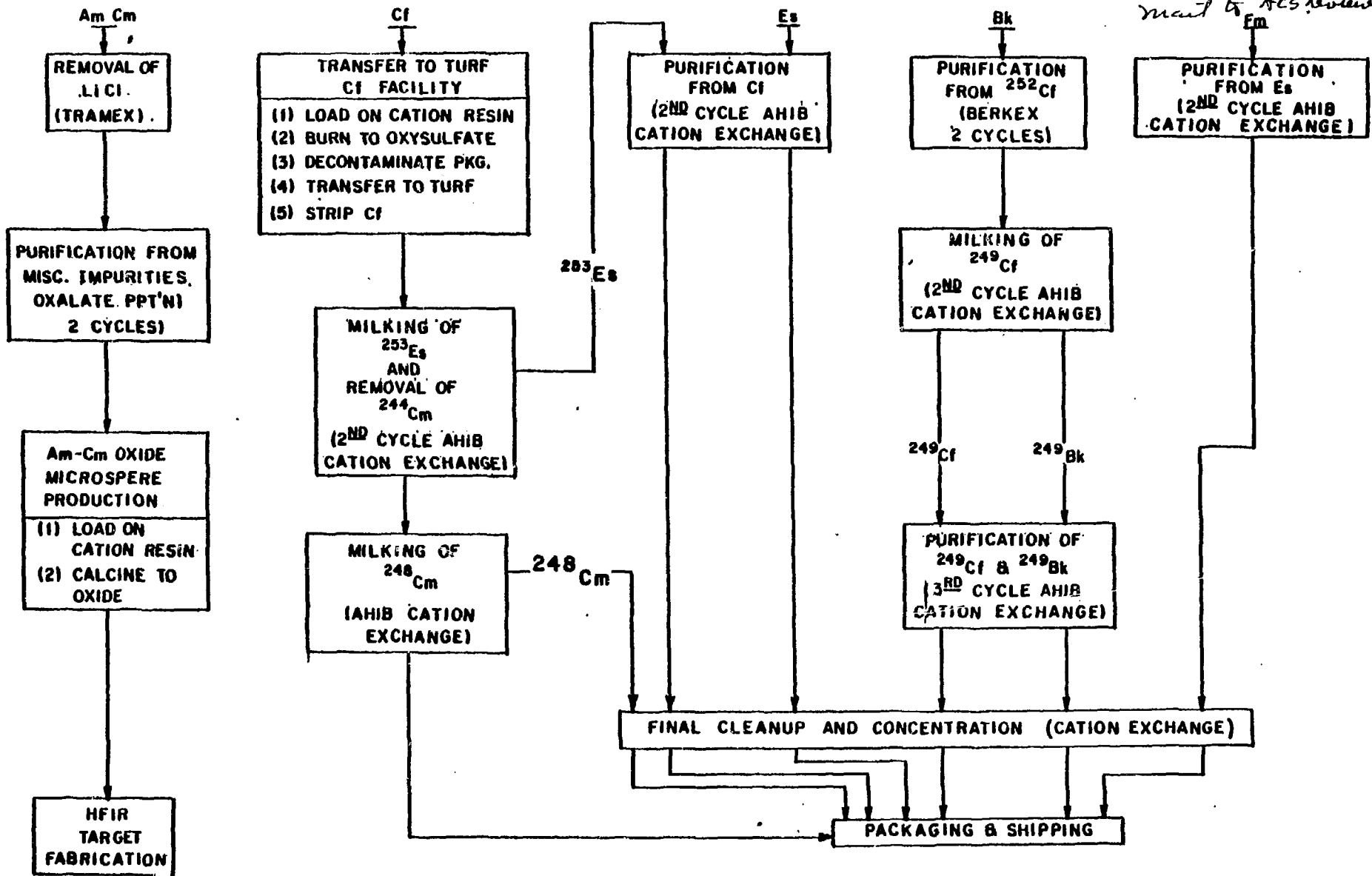


Fig. 3. Sequences of purification steps.

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50 the residual lithium) by means of two cycles of oxalate precipitation. The curium oxalate is then treated by means of a boiling HNO_3 technique to decompose oxalic acid. Following this, the curium oxide is prepared by a cation resin loading, calcination technique, which is described in another paper (12) at this symposium. The impurities remaining in a typical batch of curium oxide are all below the calculated limits for heat production and neutron absorption in the HFIR.

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35 The berkelium product fractions from the transcurium element separation runs are combined and the composite solution is concentrated and purified (13) from AHIB solution by sorbing the actinides on a cation exchange resin and stripping with 8 M HNO_3 . The berkelium is then purified from ^{252}Cf by means of one or more cycles of batch solvent extraction (Berkex) to reduce the ^{252}Cf content to less than 0.25 μg (which requires a californium DF of about 3000). NaBrO_3 is added (to 0.3 M) to oxidize Bk(III) to Bk(IV) which is extracted in a solution of 0.5 M HDEHP in dodecane diluent. The organic and aqueous phases are separated and the berkelium is stripped into 8 M HNO_3 --1 M H_2O_2 .

The berkelium, californium, einsteinium, and fermium products are then packaged and transferred from the main cell bank to other facilities in which they are purified further.

The californium is sorbed on about 0.9 ml of cation exchange resin in a platinum column, which is then calcined at 650°C (923 K) to convert the californium to the oxysulfate. (In

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50 this form, the californium can be stored for long periods and then recovered easily in a few milliliters of HNO_3 .) Then, the platinum column is put into a special package and is transferred by a pneumatic transfer system (14) to a californium purification facility in an adjoining building. A separate facility is required to minimize ^{244}Cm contamination and permit subsequent recovery in high isotopic purity of the ^{248}Cm daughter of ^{252}Cf .

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The normal processing sequence is to store the californium product about one month to permit the ^{253}Cf (a minor constituent of the californium) to decay into isotopically pure ^{253}Es . Then the package containing the column is connected to an equipment rack, and the californium is leached from the package. More than 99.8% of the californium is readily dissolved and removed from this package with about 0.1 liter of 0.5 M HNO_3 .

The isotopically pure ^{253}Es is recovered and the californium is highly purified from curium by high-pressure ion exchange using AHIB. The californium is loaded into another platinum ion exchange column and stored pending subsequent processing to recover the ^{248}Cm daughter product.

The fermium, einsteinium, and berkelium are transferred from the main hot cells and are purified further and prepared for shipment in a small hot cell and in glove box facilities that are kept free from undesirable contaminants. The chemical processing (13) involves numerous additional cycles of ion exchange purification on the micro scale.

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FIGURE TITLES

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Fig. 1. Cross section through a typical cell at TRU.

Fig. 2. Sequence of mainline steps used to process HFIR
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Fig. 3. Sequences of purification steps.

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