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ORNL-TM-850

FISSION PRODUCTS DEVELOPMENT LABORATORY

OPERATIONAL PROCEDURES

W. R. Murphy, C. L. Ottinger, and R. W. Schaich

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ISOTOPES DEVELOPMENT CENTER

FISSION PRODUCTS DEVELOPMENT LABORATORY OPERATIONAL PROCEDURES

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Isotopes Division

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ABSTRACT

This operating manual consists mainly of copies of the operating procedures currently in effect at the Fission Products Development Laboratory. Preceding the operating procedures are descriptions of the facilities that comprise the FPDL, the major chemical processes, and the basic operating practices. Much of this material has been reported elsewhere but is included here for informational purposes.

INTRODUCTION

The Fission Products Development Laboratory (FPDL) is used to process megacurie amounts of fission product radioisotopes, to convert the radioisotopes into usable source forms, and to develop and improve techniques for processing and handling large amounts of radioactive materials. Although the FPDL was originally designed for separation of fission products from wastes from uranium recovery processes, it is now used primarily for converting purified and semipurified fission product fractions received from other AEC facilities into final source forms. The main chemical processes consist of converting ^{137}Cs, received as Cs^+ adsorbed on an ion exchanger, to $^{137}CsCl$ and converting ^{90}Sr, received as solid $^{90}SrCO_3$, to $^{90}SrTiO_3$. Another large-scale process is the purification of ^{144}Ce and its conversion to $^{144}CeO_2$. However, because of its relatively short half-life (185 days), ^{144}Ce is not stockpiled as are ^{137}Cs and ^{90}Sr. Developmental work involving preparation of other fuel forms, improvement of processes, or fabrication of specific types and sizes of sources is done on a demand basis.

Facilities at the FPDL for radiochemical processing include wet-chemistry process cells, in which batch sizes of up to 400 gal can be

handled, and manipulator cells, where final processing and source fabrication are done. All process operations are carried out in heavily shielded contained cells. Auxiliary equipment includes materials-handling equipment, chemical makeup tanks, a chilled water system, and other equipment common to chemical process operations, as well as specific equipment for radiochemical processing, such as radiation detection instruments, shielded transfer casks, and special air-handling systems.

The FPDL is normally operated on a shift schedule by crews consisting of chemical operators directed by a supervisor. Shift arrangements vary with the work load. All chemical operators are trained to operate the building equipment, carry out various routine procedures, and properly use the safety and emergency facilities. Operators follow step-by-step written procedures and record appropriate data on batch data sheets. Because of the developmental nature of the operations, existing procedures are frequently changed and new procedures are initiated as needed. Procedures are prepared by FPDL technical supervisors, and before being put into operation they are reviewed by technical personnel of the Isotopes Development Center and by the appropriate ORNL review committees.

DESCRIPTION OF PHYSICAL PLANT

Building

The Fission Products Development Laboratory (Figs. 1 and 2) is a tri-level building that encloses an area of 7700 ft^2 and contains 376,000 ft^3 of free space. The first two levels, which are of standard concrete-block-side construction, consist of operating areas, service areas, offices, and other personnel access areas built around a cell block. An aluminum-sided crane bay that encloses the area over the tops of the cells forms the third level. In addition, there are several underground cells adjacent to the building, which are used for waste solution holdup and for storing solutions containing fission product fractions.

A controlled air-handling system provides ventilation and containment for the Facility. All incoming air is filtered, and all building air is exhausted through filters to the ORNL cell ventilation system. The building atmosphere is regulated at a negative pressure of 0.05 in. of water and the

Fig. 1. Fission Products Development Laboratory Building.

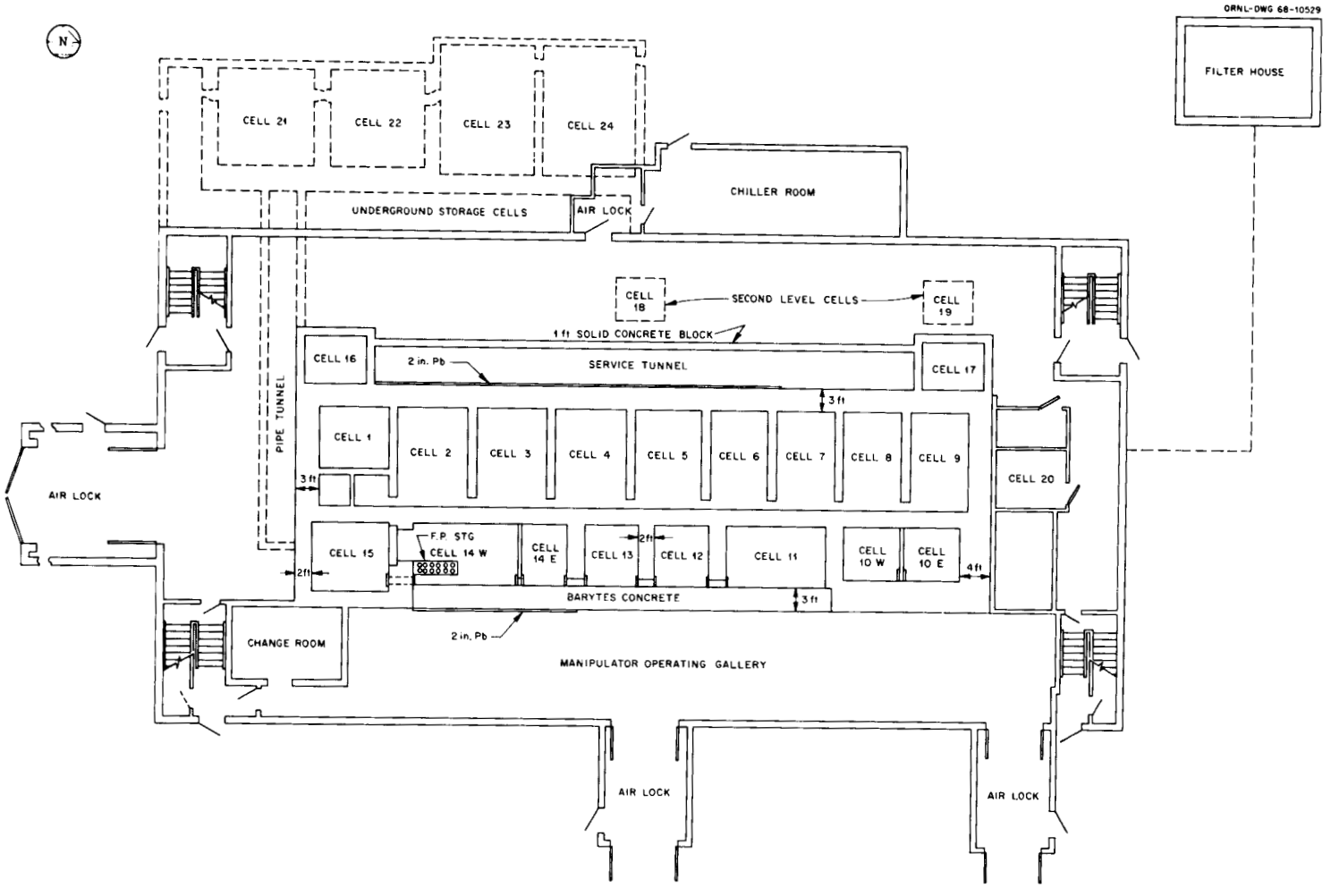

Fig. 2. Building 3517 First Floor Plan.

cells are maintained at a negative pressure of 1.5 in. of water. Interior partitions are provided to enclose all operating areas, and certain areas are mechanically cooled. Personnel entrances and equipment loading areas are airlocked to maintain atmospheric control.

Cells

Figure 2 shows the arrangement and numbering system of the FPDL cells. The main cell block consists of Cells 1 through 15. Connected to the main cell block on the first level are Cells 16, 17, 20, and the service tunnel; Cells 18 and 19 are on the second level. Four cells (21 through 24) are located underground adjacent to the building. The cell ventilation filter house is located on ground level east of the FPDL. The various types of cells available and their functions are described below.

Process Cells (1–9) contain fixed equipment and are used for remote chemical processing. The cells are totally enclosed, having no viewing windows or manipulators, and are operated from remote panelboards.

High-Level Manipulator Cells (10E–14W) are used for final processing of radioactive materials to product compounds, conversion to usable fuel forms, and packaging.

Low-Level Manipulator Cells (15, 16, 18, and 19) are used for operations which require relatively little shielding. Typical operations are sampling, decontamination of small equipment, and developmental work.

Service Cells are comprised of Cells 17 and 20, the service tunnel, and the cell ventilation filter house. These areas contain service equipment such as steam supply valves, filters, scrubbers, pneumatic transmitters, and instrument piping. Since this equipment is directly connected to the process equipment, the areas in which it is installed are treated like cells.

Storage Cells (21–24) are used for storage of radioactive solutions. These cells are like the process cells except that the equipment they contain is designed simply for storage and transfers instead of for chemical processing.

Most of the cells are lined with stainless steel for ease of decontamination. They are provided with a variety of services, such as steam,

chilled water, hot off-gas system, electrical supplies, cell ventilation, and instrumentation as required. The process cells, storage cells, and high-level manipulator cells are shielded from the nearest normally accessible area by the equivalent of 4 ft of ordinary concrete; the low-level manipulator cells and service areas have less shielding. Personnel can gain entrance to the process, storage, or high-level manipulator cells only after they remove the massive concrete top plugs. The entrances to low-level manipulator cells or service areas are equipped with interlock devices which prevent accidental entry into zones of potentially high radiation.

The Model 8 type of master-slave manipulator is used in all of the manipulator cells; the manipulators are equipped with motion locks and side-canting devices. In some cases extended-reach or heavy-duty manipulators are used. The slave ends of the manipulators are booted in plastic to protect them from contamination. The viewing windows in Cells 10E, 10W, 15, and 16 are glass tanks containing zinc bromide solution for shielding; those in the other manipulator cells are of high-density glass.

Process Equipment

Permanently installed equipment in the FPDL provides for the following operations:

Elution of ion exchanger beds
Precipitation-centrifugation
Precipitation-filtration
Crystallization-filtration
Evaporation
Batch solvent extraction
Calcination and sintering
Hydraulic processing (hot and cold)

In addition to these operations, essentially any unit operation or process can be carried out on a relatively small scale in the manipulator cells with temporary equipment.

Three of the process cells (2, 3, and 4) contain precipitators, centrifuges, and holdup tanks. These cells are arranged so that a series of operations can be carried out on one batch of solution, with the centrifugate from one operation being transferred to the precipitator in the next cell. However, each cell is equipped to handle all the processing operations on a single batch of solution.

In the crystallization cells (5–8) the equipment provides for repeated crystallization, filtration, and dissolution of crystal beds. The mainstream crystallizers have a 400-gal working capacity, and the concentrate crystallizers can process 100-gal batches. The piping is designed so that all filtrates can be recycled from the concentrate crystallizers, as well as from the manipulator cell process vessels, to the mainstream system.

The solvent extraction cell (9), which is used specifically to extract cerium from a solution of mixed rare earths, contains two batch extractors, a precipitator, and associated tanks. There is no solvent recovery equipment since solvents are not re-used in the FPDL process. Direct piping to a cask-loading cell permits rare earth solutions to be loaded for transfer to ion exchange equipment at another Isotopes Division facility.

Feed solutions and concentrate fractions are stored in tanks in underground cells. The tanks are equipped with cooling fins, and the cells are partially filled with water. Two of the precipitator cells (3 and 4) are also used for storage of ^{90}Sr feed solution.

Three of the manipulator cells (11, 13, and 14E) can also be used as precipitator cells; in these cells the precipitators can be connected to vacuum receiver vessels, with the precipitates being collected on filters and then handled by the manipulators. Other equipment in the manipulator cells includes furnaces, blenders, and hydraulic presses, as well as standard laboratory-sized general equipment. The two small manipulator cells on the second level (18 and 19) are used for process control. They contain recirculating sampler lines for drawing representative samples from the process vessels. If an analysis is required, the samples can be loaded for transfer to an analytical facility.

All tanks and process piping in the FPDL are of welded stainless steel. The tanks are equipped with liquid-level indicators, temperature indicators, and other instrumentation necessary for control of the processes. Most of the process operations are carried out from graphic panelboards. All aqueous solutions are transferred by steam-jet vacuum or by gravity flow. Organic solutions are transferred by pumps or airlifts.

Auxiliary Equipment

Batch makeup tanks for process chemicals are located on the second level of the building. A makeup and storage area for large volumes of process chemicals is located outside the building. Chilled water for cooling the process vessels is provided by two compressor units on a recirculating water loop. A 20-ton bridge crane is used to handle shipping casks and to remove the top blocks from the cells when required.

In-Cell Maintenance

The FPDL was designed to be directly maintained. Before any equipment can be repaired, replaced, or revised, the equipment and the cell in which it is located must be decontaminated. The process cells are decontaminated by first circulating decontamination agents through the tanks and the pipes in a definite pattern and then opening the cells and washing the surfaces of the vessels and the cell walls. If necessary, shielding is then used to reduce the radiation level to acceptable limits. Decontamination of the manipulator cells is the same as for other cells except that the contaminated solid waste is removed for land burial.

Some semiremote maintenance can be done. For example, agitator mountings on process vessels are arranged so that the agitators can be disconnected and removed from the cell without the necessity for cell entry. In the manipulator cells the equipment is positioned for easy access by the manipulators, and minor repairs can be made by completely remote methods.

CHEMICAL PROCESSES

Cesium-137

The flow sheet for the ^{137}Cs process is shown in Fig. 3.

Receipt

Feed for the ^{137}Cs process is shipped from Richland, Washington, in Shielded Transfer Tanks (STT's) which contain 1 ton (400 gal wet volume) of an aluminosilicate ion exchanger (Decalso). These tanks hold 500 gal, are shielded by 3.5 in. of lead, and weigh 19 tons. A new model of STT's, which contains 110 gal of AW-500 ion exchanger, has recently been put into service. Both models of STT's normally transport ~50,000 Ci. The STT's

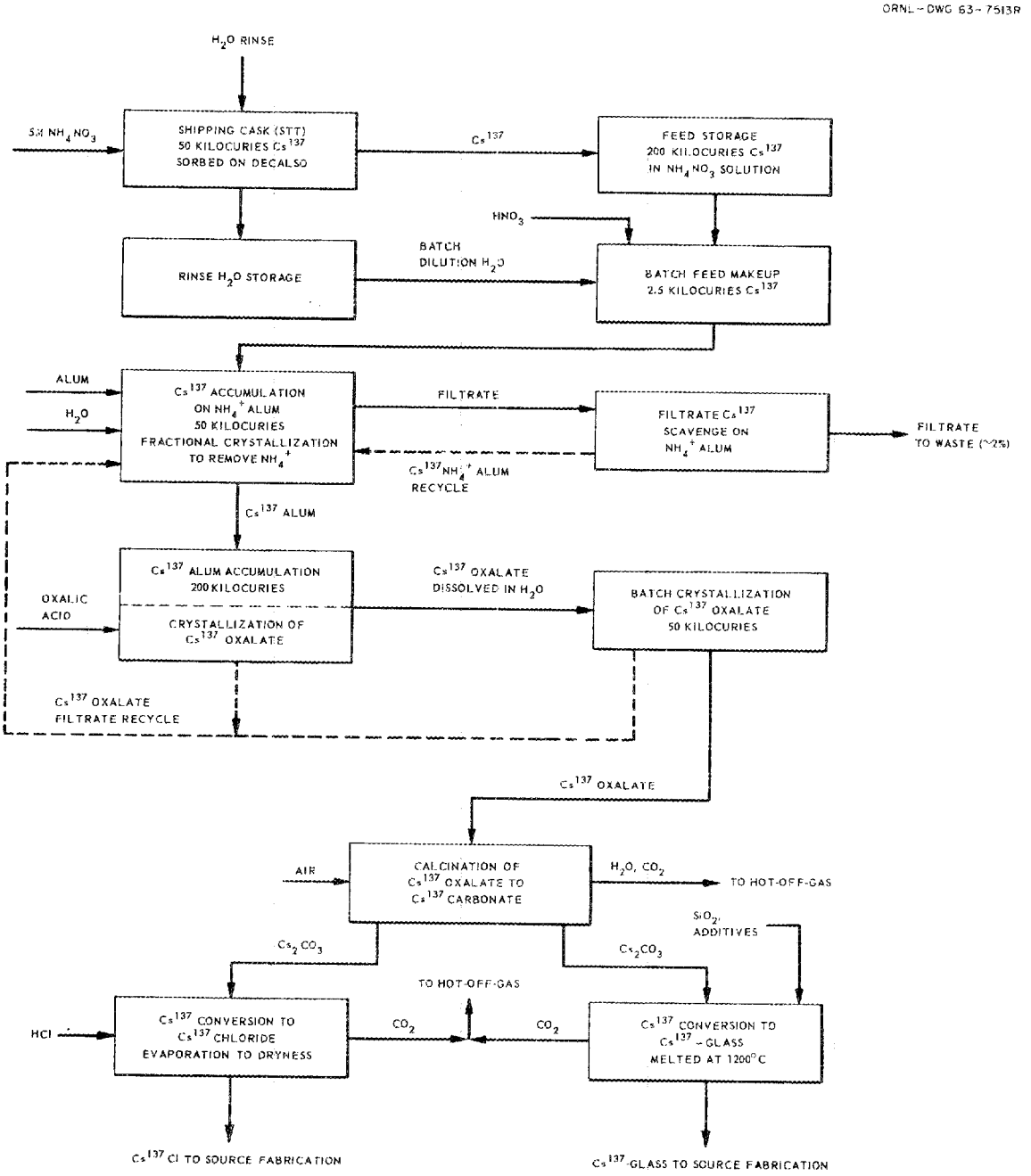

Fig. 3. FPDL ^{137}Cs Processing.

are enclosed in fire shields on specially constructed railcars, which carry two STT's each. Shipment requires from five to ten days, and the railcars are delivered to the Oak Ridge Gaseous Diffusion Plant rail siding. There the fire shields are removed, and the STT's taken off the cars and delivered to ORNL by truck. All STT's are vented soon after arrival; then, depending on length of storage, temperature, etc., they are vented periodically before elution. Elutions are done as soon as operational requirements permit.

Elution

The ^{137}Cs is eluted from the ion exchanger by passing 5 M NH_4NO_3 at 80°C through the bed. Before the elution is started, the bed is preheated with 500 gal of water at 80°C. Elution is continued until approximately 95% of the ^{137}Cs has been removed. The ion exchanger bed is then rinsed with cold water, and the STT is prepared for shipment back to the supplier.

Although the preheat water contains only trace amounts of ^{137}Cs and the rinse water contains ~1% of the ^{137}Cs that was on the ion exchanger, these solutions are usually reserved for use in diluting the ^{137}Cs feed solution during the crystallization process. The eluted ^{137}Cs solution is sampled and held in storage tanks until it can be processed.

Crystallization

Feed preparation for crystallization is done batchwise. The starting solution, usually about 75 gal, is acidified to 1 M in HNO_3, digested, and diluted 3:1 with water. The dilution is necessary because the high initial ammonium ion concentration of the feed would cause high ^{137}Cs losses.

The method for accumulating ^{137}Cs is by co-crystallization with ammonium alum. The crystal bed size is controlled at 40 ± 10 gal. The filtrate from the first crystallization contains ~10% of the ^{137}Cs content of the primary crystallizer. The filtrate is transferred to another crystallizer, where it is scavenged by an identical crystallization procedure. The filtrate from the scavenge crystallization may also be scavenged, by the identical process, if the ^{137}Cs content is sufficiently high to warrant it. When certain limits of ^{137}Cs in the scavenge crystallizers are exceeded, the crystal beds are reduced by water crystallization and then dissolved and transferred back to the primary crystallizer.

When ~50,000 Ci of ^{137}Cs have been accumulated in the primary crystallizer, the crystal bed is reduced by water crystallization. The crystal bed is then dissolved in 1 M HNO_3, and the solution is transferred to the concentrate crystallizer system. Normally, four cycles of accumulation, crystal bed reduction, and transfer are done, resulting in an accumulation of ~200,000 Ci of ^{137}Cs in the concentrate crystallizer system. The ^{137}Cs concentrate at this point still contains traces of rubidium and ammonium alums. These contaminants are removed by repeated water crystallizations to yield a purified ^{137}Cs alum fraction. Filtrates from the concentrate ^{137}Cs crystallizations are recycled to the primary crystallizer.

Oxalate Processing

In the concentrate crystallizer, cesium tetraoxalate is crystallized from the alum system by the addition of solid oxalic acid. After the cesium tetraoxalate has been filtered out, the filtrate is recycled back to the primary crystallizer. The cesium tetraoxalate crystals are dissolved in water, and this solution is transferred in batches of ~50,000 Ci each to a final crystallizer in a manipulator cell. Another oxalate crystallization is done on each batch, and the resulting crystals are collected on filters. By manipulator operations the crystals are loaded into a furnace, where they are dried and then calcined to form $^{137}Cs_2CO_3$.

$^{137}CsCl$ Preparation

The $^{137}Cs_2CO_3$ is converted to $^{137}CsCl$ by the addition of a slight excess of HCl to a solution of $^{137}Cs_2CO_3$. The resulting solution is filtered, evaporated to dryness, and baked at 350°C for 4 hr to remove the last traces of HCl. The dry ^{137}Cs product is stored in stainless steel cans in lots of ~20,000 Ci. All the $^{137}CsCl$ preparation operations are done in laboratory-scale equipment which is operated by manipulators. The powder can be assayed by calorimetrically measuring the heat output of a known weight of powder, or a known weight of powder can be dissolved for analysis.

Pellet Fabrication

Pellets of $^{137}CsCl$ are formed by pressing the dry powder in tool-steel dies. Cylindrical pellets of various sizes can be fabricated. One of the pellet configurations most often used is rectangular, usually 1 x 1 x 1/4 in.

in size. When pellets are prepared before they are ready to be loaded into sources, they are stored in cans similar to powder storage cans.

Strontium-90

The flow sheet for the ^{90}Sr process is shown in Fig. 4.

Receipt

The feed material for the ^{90}Sr process is purified $^{90}SrCO_3$. It is shipped from Richland, Washington, as dry solid on specially designed filter shipping casks, designated HAPO-IB and HAPO-II, which differ only in size. The HAPO-IB casks weigh 20 tons and can transport 500,000 Ci of ^{90}Sr, whereas the HAPO-II casks weigh 9 tons and can transport 200,000 Ci of ^{90}Sr. The casks are shipped by rail to the Oak Ridge Gaseous Diffusion Plant and are then delivered to the FPDL by truck.

Unloading

The HAPO cask is put in the unloading cell and is vented to off-gas. While the cask is still vented, the short leg of the cask is connected to a steam-jet discharge line and the long leg is connected to a water feed tank. The vent is then closed and the cell is sealed. A steam jet is used to create a vacuum in the cask and thus pull water from the feed tank through the cask. The filter cake of $^{90}SrCO_3$ is broken up, and the slurry of $^{90}SrCO_3$ is discharged into a collection tank. After most of the $^{90}SrCO_3$ has been removed, the cask is washed with weak HNO_3. The HNO_3 is discharged into the collection tank containing the $^{90}SrCO_3$, and the ^{90}Sr is converted to $^{90}Sr(NO_3)_2$. A water rinse completes the unloading operation, and the cask is prepared for return shipment.

Preparation of $^{90}SrTiO_3$

Batches of 40,000 Ci of ^{90}Sr are converted to $^{90}SrTiO_3$ in a manipulator cell. The $^{90}Sr(NO_3)_2$ feed solution is adjusted to pH 3 to 5 with NH_4OH; then a slurry of $(NH_4)_2CO_3$ and TiO_2 is added. The system is filtered and the $^{90}SrCO_3$-TiO_2 mixture is calcined to yield $^{90}SrTiO_3$. The dry $^{90}SrTiO_3$ is assayed by calorimetry and then stored in stainless steel cans.

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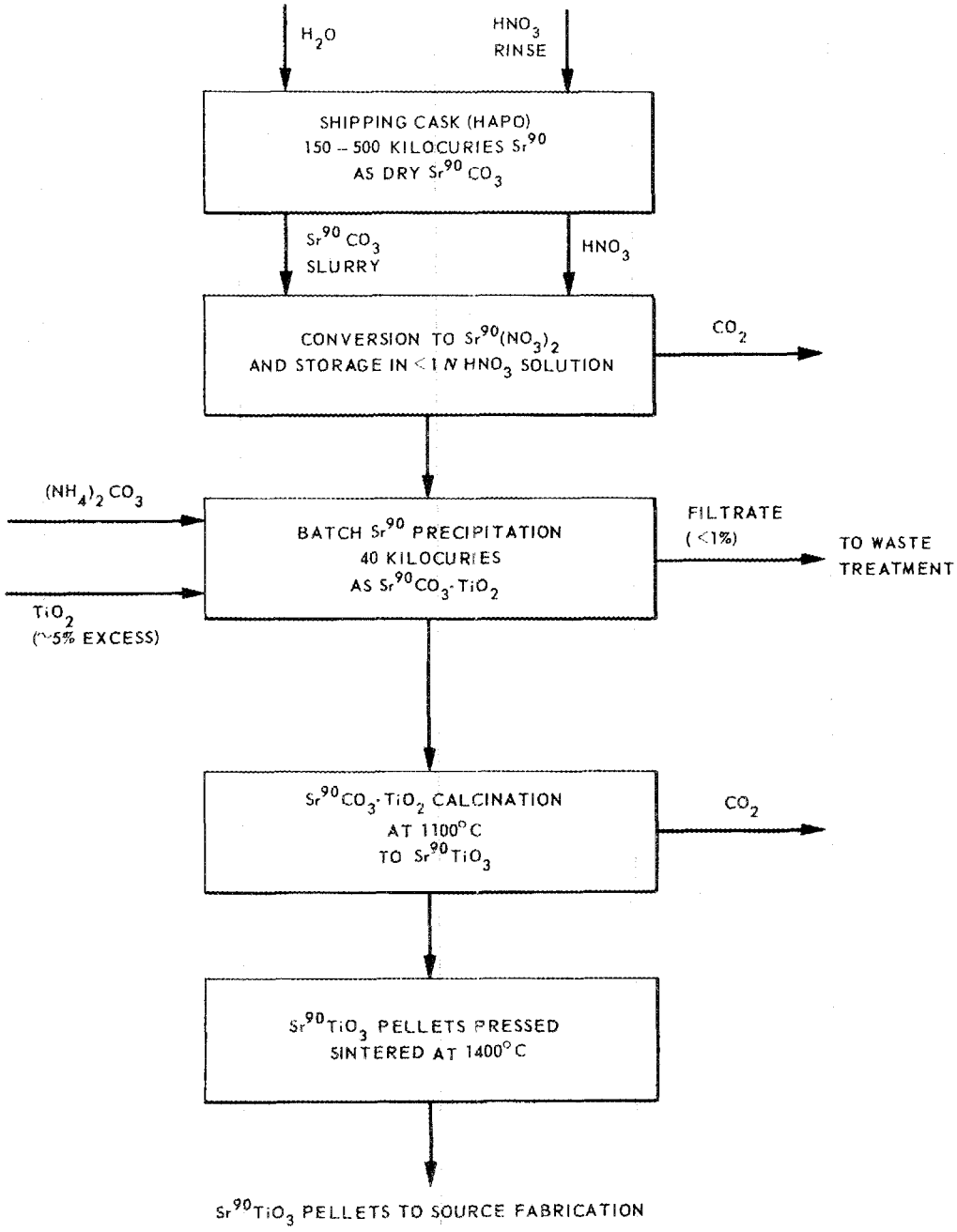

Fig. 4. FPDL ^{90}Sr Processing.

Pellet Fabrication

Two types of $^{90}SrTiO_3$ pellets can be fabricated: cold-pressed and sintered pellets and hot-pressed pellets. The cold-pressed pellets are made by compacting calcined $^{90}SrTiO_3$ in tool-steel dies and then sintering them to improve their density. The hot-pressed pellets are made by loading the $^{90}SrTiO_3$ into graphite dies, placing the dies in the cavity of a furnace, heating the furnace while the cavity is purged with argon, and hydraulically pressing the powder while it is hot. Finished pellets are weighed, measured, and in some cases individually assayed by calorimetry. Pellets are usually fabricated just before they are encapsulated into sources but some may be stored temporarily. Since the size, curie content, and required densities of pellets vary according to the particular project, pelletization procedures are rather general.

Cerium-144

The flow sheet for the separation and purification of ^{144}Ce is shown in Fig. 5.

Receipt and Unloading

Dry mixed rare earth sulfates containing ^{144}Ce, ^{147}Pm, and stable rare earths are shipped from Richland, Washington, on casks like those used for shipping ^{90}Sr. The shipping, cask handling, and cask unloading techniques are identical to those used for ^{90}Sr.

Separation

The rare earths are concentrated by precipitation of the rare earth oxalates. The oxalates are dissolved in HNO_3 to prepare feed for the solvent extraction separation. Stable yttrium (and ^{91}Y if present) is separated by extraction into 0.5 M di(2-ethylhexyl)phosphoric acid (D2EHPA); the organic phase is discarded. Cerium is oxidized to Ce(IV) by $KMnO_4$, and the Ce(IV) is extracted into 0.5 M D2EHPA. The aqueous phase from this extraction contains ^{147}Pm and stable rare earths. An oxalate precipitation is performed on the aqueous phase. Then the oxalates are dissolved in HNO_3 to yield a rare earth solution, which is transferred to an ion exchange separation unit at another Isotopes Division facility. The ^{144}Ce is backextracted from the organic phase by reducing

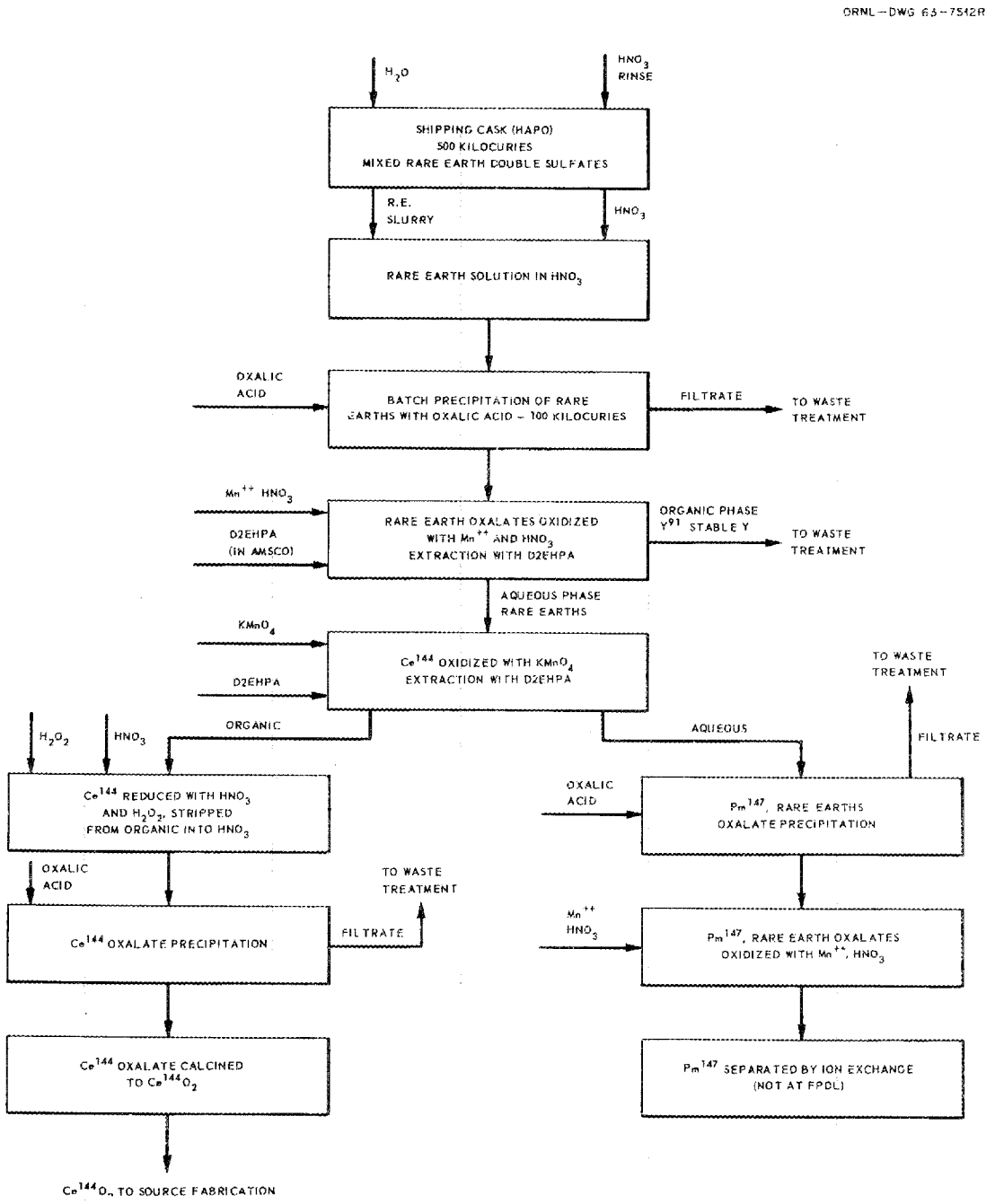

Fig. 5. Flow Sheet for Separation and Purification of ^{144}Ce.

the Ce(IV) to Ce(III) with H_2O_2 and stripping with 2 M HNO_3. Other extraction procedures can be used if the feed solution contains significant amounts of interfering ions, such as corrosion products.

Final Processing

The HNO_3 solution of purified ^{144}Ce is transferred to a manipulator cell, where an oxalate precipitation is done. The cerium oxalate is calcined to $^{144}CeO_2$, which is then pressed into pellets for source fabrication. The techniques for handling $^{144}CeO_2$ are similar to those used for $^{90}SrTiO_3$.

Source Fabrication

Encapsulation of products into sources or shipping containers is done as required. Sizes of packages vary from as little as 5 Ci to more than 100,000 Ci in individual containers. The methods of encapsulation are similar for packages of all sizes. The most commonly used encapsulation materials are type 316L stainless steel for $^{137}CsCl$ sources and Hastelloy C for $^{90}SrTiO_3$ sources, but other materials can be used. Sources are always closed by welding, and two welded containers are usually required. Welding methods are developed for each specific source configuration. The necessary quality control tests vary from source to source. Some typical tests are vacuum leak tests, thermal cycling tests, thermal output measurements, and contamination checks. Sources are usually made by loading pellets of the radioactive fuel material into the containers, but loose powder is sometimes used. Bulk powders are shipped in welded containers made of type 304 stainless steel. Since there are no standard sources, there are no standard operating procedures for source fabrication.

Developmental Processes

Special projects are undertaken by the FPDL in its role as a development laboratory. These projects may be adaptations of existing processes, as in the preparation of special grades of $^{90}SrTiO_3$ powder or the conversion of ^{137}Cs to $^{137}Cs_2SO_4$, or projects that can take advantage of some particular equipment feature, as in the loading of radioisotope-powered thermoelectric generators. The various special projects may vary from

those which require only a few man-hours of time and no special equipment to those which last for several months, use a major fraction of the FPDL manpower, and necessitate major equipment revisions. Examples of some of the special projects that have been carried out are the separation of enriched uranium from reactor fuel (in special equipment in a manipulator cell), conversion of ^{137}Cs to glass source forms, and preparation of the fuel for a series of ^{90}Sr-powered generators.

TYPES OF PROCEDURES

The procedures used at the FPDL fall into three categories: general procedures, safety and health physics procedures, and process procedures. Written procedures are prepared for all operations before the operations are started. These procedures are reviewed by technical personnel of the Isotopes Development Center, by the proper administrative and supervisory personnel, and when appropriate by various ORNL review committees. The reviewing personnel evaluate the procedures with respect to safety, compatibility with other FPDL operations, and other considerations.

The procedures for special projects are written like those for routine projects. When the special project is brief or when full-time technical supervision is needed, the procedures are handwritten. At other times existing procedures are modified to cover the special project. When a project is to be of long duration or when it has special safety aspects, a full formal procedure is prepared and the operating personnel are trained in its use.

Procedure Manuals

Written procedures are kept in manuals that are located at the various operating areas for use by the operators. Two master copies are kept for use by the FPDL supervisory personnel. The manuals are arranged so that procedures concerning a certain process stream are located together. For example, all the process procedures for ^{137}Cs crystallizations are in one manual, and no other procedures are included. There are sufficient copies of each manual for each operator working on a given operation to have one.

A supply of blank pages entitled "Special Instructions" is kept with the operators' manuals for use by the supervisors. These pages are used for temporary notes concerning specific steps or minor changes in particular operations and are inserted in the manual opposite the procedure involved. When the process or operation is finished, the "Special Instruction" sheets are removed from the manual and filed.

Procedures are written in step-by-step order, and the operating personnel are required to follow them exactly. Any unauthorized deviation from a written procedure is considered grounds for disciplinary action. Significant data are recorded on batch data sheets, which then become part of the permanent process records.

Process vessels, lines, and valves in the FPDL system are numbered and coded. In the preparation of procedures, however, the coding system is used only for vessels; the identification of the valves and lines is descriptive. The process vessel identification follows a code in which a "P" tank is always a precipitator, a "G" tank is a centrifuge, a "C" tank a crystallizer, etc. These codes are marked on the graphic panelboards, and all lines, valves, and services are also shown. In performing an operation the operator reads the description of the step in the procedure; then he performs the specified operation by locating the proper controls on the graphic panel and taking the actions required in the procedure.

General Procedures

The general procedures cover the operation of the auxiliary equipment, use of special manipulator cell equipment, chemical makeups, and similar operations. Some of the general procedures, such as the "Calorimeter Operating Procedure," are used in conjunction with operational procedures; others, such as the "Demineralizer Regeneration Procedure," are routine and are done more or less independently of the process operations. General procedures are carried out either on a predetermined schedule, as instructed by supervision, or as called for in another procedure.

Process Procedures

All process operations at the FPDL are covered by detailed procedures; these operations include any work done on radioactive materials from the time they are received as feed until the final products are shipped. For the wet-chemistry processes, most procedures contain a chemical makeup section, a series of operating sections, and a section concerning the transfer of the various streams to other processes. Most processes can be handled by one operator, but some require two or more operators to work together.

Procedures for operations that are conducted in manipulator cells are necessarily more descriptive than those for work performed at panelboards. However, the manipulator cell operations can be controlled better because they can be followed visually.

Safety and Health Physics Procedures

Safety and health physics procedures in force at the FPDL include safety regulations governing the use of safety equipment, emergency procedures, procedures for working in hazardous areas, and zoning regulations. These procedures are not used operationally in the sense of being performed step by step, but rather are used to define the actions necessary in certain instances. All personnel who work at the FPDL are required to be thoroughly familiar with these regulations. In addition to the standing safety regulations, specific procedures are issued to cover hazardous aspects of particular operations, especially in new projects. Also, the operating procedures include steps which deal with the safety aspects of the system.

OPERATIONS

Shift Operations

The FPDL normally operates on a schedule of two shifts five days a week; each operating crew consists of seven chemical operators directed by a supervisor. Additional chemical operators and supervisors are assigned to the straight-day shift. The shift supervisors make work assignments, maintain records, train personnel, and perform other duties required by

direct supervision. When new procedures are to be tested, additional technical personnel will frequently be assigned to follow a specific operation. Shift supervisors work under the direction of the FPDL supervisor, and directions are given in an instruction log book. Changes in existing procedures are entered in a master procedure manual; then the shift supervisors make the appropriate revisions in the manuals used by the operating personnel. The operations conducted during each shift are described in a supervisor's log book, which is reviewed by the next shift supervisor.

Processing Schedules

All FPDL operations are carried out on a "campaign" basis, with the length of the operation varying from a few hours for a test batch to many weeks for a major source fabrication. During full-time ^{137}Cs operations the processing rate is about 30,000 Ci per week and the annual capacity for $^{137}CsCl$ production is ~1,000,000 Ci. The processing rate for $^{90}SrTiO_3$ production is ~100,000 Ci per week and the annual output is ~2,000,000 Ci. Annual outputs are controlled primarily by the availability of feed; those quoted above reflect the current shipping practices based on the number of casks and the loading per cask. Since ^{144}Ce is processed strictly on a demand basis, its annual output cannot be defined, but individual shipments of 500,000 Ci can be made and as much as 100,000 Ci per week can be processed.

Processes and equipment at the FPDL are arranged for batch processing. The chemical process systems can be shut down within a matter of minutes, then be restarted with little effect on the operations other than loss of time. This allows considerable flexibility of operations and permits manpower to be allotted to projects of immediate priority.

The rate-controlling operations for ^{137}Cs, ^{90}Sr, and ^{144}Ce are cesium carbonate preparation, strontium titanate precipitation, and cerium oxalate precipitation, respectively. Since the equipment involved for each of these operations is identical, all three final processing cells can readily be used to prepare the same product should its demand increase, provided of course that the amount of feed available is correspondingly increased. In this fashion the annual output of a single product could be increased to more than 5 MCi.

Operator Training

The training of chemical operators is conducted by the FPDL shift supervisors. The basic training period for a new operator includes instructions in the arrangement of the equipment and its operation, in the various general procedures, and particularly in the safety aspects of the Facility. During this period the operator learns the tank numbering system, the cell identifications, and the terminology of the written procedures. He also receives training in the use of radiation-detection devices and other safety equipment. After this basic period the operator is assigned to one of the shift crews, where he begins to learn the specific operational procedures. Several months of training, varying somewhat according to the individual, are required before a new operator can confidently conduct the routine processes.

When new procedures are adopted or when special projects are begun, training of the operators is considered an important part of the project. First, only the operators needed to conduct the work are trained. Then, as the project progresses, other personnel are trained. For short-term projects it is impossible to train the entire operator force, so selected individuals are used for the time required.

Since the FPDL is a development laboratory and as such requires frequent changes in procedures, all personnel must be constantly attentive to revisions in the procedures. The ability to adapt to varying conditions is one of the most valuable traits of any qualified worker. Regardless of the number and detail of procedures, the safe and efficient operation of the Facility depends finally upon the intelligence, training, and interest of the individual worker.

PROCEDURES

This section includes copies of all the procedures that were in effect at the time this report was prepared. Since the FPDL is essentially a developmental facility, procedures will be changed as necessary to adapt to changing requirements, to improve existing processes, or to investigate new processes.

The first 21 procedures are general procedures, the next 19 are process procedures, and the last 4 are safety and health physics procedures. Batch data sheets, inventory forms, and similar records are included with the appropriate procedures. Some data sheets, such as the "Furnace Record," are used with several procedures. In such cases the data sheet is included with only the first procedure to which it applies. (Standard ORNL forms such as Radiation Work Permits, Materials Transfer Tags, etc., are not reproduced in this manual even though they are frequently used.) The procedures are reproduced as they appear in the operating manuals, but without any process notes or special instructions. The blank spaces in the various procedures indicate that conditions are variable and subject to frequent change. The values for the blank spaces are given on "Special Instruction" sheets facing the procedures.

The titles of the procedures and the pages on which they appear are listed on the following pages.

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FISSION PRODUCTS DEVELOPMENT LABORATORY

GENERAL PROCEDURES

I. General

A. Policy

FPDL operations are conducted in accordance with established ORNL policies and procedures. All personnel are expected to comply with all ORNL rules and regulations as covered in the Standard Practice Procedures and other official publications.

B. Work Assignments

1. Operators will normally be assigned to a specific crew and will receive their assignments from the supervisor of that crew. Occasionally an operator will be assigned to work for another FPDL supervisor or for other Isotopes Division personnel attached to the FPDL for special projects.

2. Operators are required to be at their work location or assembly point, ready to work, at the beginning of their scheduled shift. Depending on the status of operations, specific operators may be given assignements which will last for several shifts, and they will report to the appropriate work location. Operators not having such assignments will assemble at a location specified by their supervisor.

3. Lunch periods and rest breaks will be arranged by supervision. There is no fixed schedule for these periods since operations vary from day to day. The duration of these periods is established by company policy.

4. Except in emergencies, operators will not leave their work location without approval of supervision.

C. Clothing and Equipment

1. The normal work dress for FPDL operations includes khaki work clothes or C-zone coveralls, safety shoes, safety glasses, pencil meters, and film badges.

2. During shift change periods and in some other instances, supervision may arrange alternate clothing changes for relief purposes. In these cases an operator may work in personal clothes - <u>in noncontamination areas only</u> - for short periods.

3. Additional protective clothing and/or equipment may be required by procedures or by supervision. This equipment is either stocked in regular locations or issued separately.

D. Safety

1. Performing all operations in the safest possible manner is a responsibility shared by all FPDL personnel. In addition to the hazards involved in any chemical plant operations, the handling of large amounts of radioactive materials presents special problems at the FPDL. Actions that create a hazard to an individual, to other personnel, to the environment, or to the facility are not tolerated.

2. All personnel are expected to be thoroughly familiar with the ORNL and FPDL safety regulations and to comply with them in all particulars.

3. Operators will report unsafe conditions to supervision as soon as they note them.

4. Violation of safety regulations is considered to be grounds for disciplinary action.

E. Housekeeping

1. All personnel are expected to maintain acceptable levels of housekeeping in their work locations and to cooperate in the maintenance of good housekeeping in the building generally.

2. Special attention is to be given to housekeeping practices in the lunchroom and the change room to prevent these areas from becoming cluttered.

3. Good housekeeping practices are to be applied to the interiors of manipulator cells as well as to the other areas of the building.

II. Operational Procedures

A. Policy

1. FPDL operations are covered by specific written procedures to the greatest practical extent. For work not covered by standard procedures, handwritten or verbal instructions are issued by supervision.

2. Any deviation from an established procedure without specific and direct approval of supervision is considered grounds for disciplinary action.

B. Procedure Form

1. FPDL standard procedures are of three types: process procedures, general procedures, and safety procedures. These procedures are distributed in several manuals, arranged according to the subject operation. Copies of these manuals are located as necessary in the operating areas.

2. Procedures are written in step-by-step orders, and the sequence of operations must be followed exactly.

3. Procedure manuals are arranged so that a blank page faces each printed page. Notes, changes in procedures, chemical additions, and other information may be entered on these blank pages by the building supervisor. Such entries are considered part of the procedures.

4. Procedures are written on the assumption that the operator is completely familiar with the nomenclature of the vessels, lines, and in-cell equipment. If a step in a procedure is not understood, the operator will contact supervision before proceeding.

5. If for any reason a step in a procedure cannot be done as described, the operator will contact supervision before proceeding.

C. Data Sheets and Log Books

1. For procedures that require data to be kept, separate data sheets are used. Blank data sheets are issued as needed. Operations are not to be started until the appropriate data sheet has been issued.

2. Data will be entered as it is obtained, not after the procedure is completed.

3. All data sheets must be dated. Run numbers and other information will be issued by supervision.

4. Data sheets must be filled out completely. If a particular entry cannot be made, the reason why it cannot will be noted on the sheet.

5. Completed data sheets will be turned in to supervision.

6. Log books are kept at the operating areas for use by operators. A brief description of the operations conducted during the shift will be entered in the log, along with notes and observations.

D. Routine Procedures

1. Shift checks, daily checks, inventories, and equipment checks are taken routinely. Supervisors will assign these checks.

2. Routine check sheets must be filled out completely. Any deviation from the limits shown on the sheets will be reported to supervision.

3. The routine checks that are made each shift are those shown on the "Shift Check Sheet," which is filled out at the beginning of the shift, and the "Shift Tank Inventory," which is taken near the end of the shift.

   a. The data taken for the "Shift Check Sheet" includes checks on the off-gas system, the cell ventilation system, the ^{90}Sr solution storage system, and certain items in the manipulator cells. After the data have been recorded, the operator will report to the supervisor, who will review the data and take any corrective actions indicated.

   b. The completed "Shift Tank Inventory" will be turned in to the supervisor, who will review the data and compare the inventory with the one from the previous shift. Any changes not related to current processing will be noted and investigated.

4. The routine checks that are made on a daily basis (usually on the day shift) are those shown on the "Daily Check Sheet" and the "Daily Chemical Inventory," both of which are taken at the beginning of the shift. The completed forms will be turned in to the supervisor, who will review them and take any actions indicated.

5. The weekly routine checks are usually made on Friday during the day shift. These checks are the "Weekly General Supplies Inventory" and the "Weekly Check List." The items on the "Weekly Check List" are related to the building emergency system and are checked as shown in Steps a through g below. The locations of the various items are shown on drawings in the procedure book.

   a. Emergency lanterns are tested by turning on the test switch to see if the light operates, and then turning off the switch.

   b. The emergency siren is tested by activating the "building evacuation" switch in the shift supervisor's office. The siren should be left on just long enough for the sound to be heard and turned off before it reaches full volume.

c. The entrance door lock system is checked by activating the "emergency door lock" switch located in the shift supervisor's office. While the switch is on, each personnel entrance door is checked to see if the electrical locking pin is engaged, and the crash bar on each door is checked to see if the door can be opened from inside.

d. Each airlock interlock system is checked by opening the inside airlock door and checking that the electrical lock on the outside door is engaged. While the inside door is open the outside blue signal light should be on. The inside airlock door is then closed and the outside door opened to check the electrical lock and signal light on the inside door.

e. The emergency assault-mask boxes are checked to see if they are completely filled. Fresh, plastic-sealed, assault masks will be put in any slots that are empty.

f. The emergency cabinets are checked and restocked according to the list posted on the cabinet. The cabinet doors are then closed and taped with masking tape.

g. The building containment system is checked only after contacting the supervisor, who will announce the check over the paging system. The supervisor will activate the containment system manually and reset the system after the final pressure and the time required to reach final pressure have been recorded.

h. After all of the checks have been made, the operator will turn the completed check list in to the supervisor, who will review it and take any corrective actions indicated.

E. Basic Operating Rules

NOTE: The rules listed below are applicable to most panelboard and/or manipulator cell operations. Many of these rules are also included in specific procedures.

1. Answer all alarms immediately. Take the necessary corrective action or report to supervision immediately.

2. Keep the safety caps on all valve switches except when operating the switch. Never operate a switch that is tagged "Do Not Operate." Replace the safety cap when a switch is put to the off position.

3. Observe the following jet-operation rules:

   a. Double-check flow direction before operating.

b. Check the volume of the tank to which solution is being transferred. Be sure that enough free space is available.

c. Do not jet solutions with temperatures above 70°C.

d. Do not jet solution into a tank if the tank temperature is greater than 70°C.

e. After jetting has started, check that the solution is going to the proper tank.

f. Never leave an operating jet unattended.

g. Watch the off-gas on tanks being jetted into and if it falls below 2 inches, stop jetting.

h. Never jet from two different jets into the same tank.

i. Check the temperature on the jet discharge. A sudden increase in temperature shows that solution is not going through the jet.

j. Close the jet immediately after the solution has been transferred.

k. Never jet to a centrifuge that is not running.

4. Label all tanks as to volume, contents, and date after making transfers. When the solution can be identified by a sample code, include the sample code on the label.

5. Identify all radioactive material in a manipulator cell by means of notes attached to windows. This is especially important at shift change.

6. Report any equipment failure to supervision immediately.

7. In case of a manipulator arm failure or boot failure, make the minimum amount of motion with the arm. Then lock out or rope off the arm and report to supervision.

FISSION PRODUCTS DEVELOPMENT LABORATORY

SPECIAL INSTRUCTIONS

Procedure _____

Page _____ Section _____

Date of Special Instructions _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

SHIFT TANK INVENTORY

Operator_____ Shift_____ Date_____

UNLOADING AND STORAGE SYSTEM				CESIUM-137 SYSTEM			
Tank	Batch	L.L.	Remarks	Tank	Batch	L.L.	Remarks
P12				W19			
F12				W20			
H12				S122			
F421				P45			
F521				H45			
F621				E16			
F821				C17			
F1021				C27			
F1121				C38			
S223				C48			
S523				P713			
S424				P114			
S324				R18			

STRONTIUM-90 SYSTEM				RARE EARTH SYSTEM			
Tank	Batch	L.L.	Remarks	Tank	Batch	L.L.	Remarks
P23				P89			
F23				H89			
H23				N19			
P34				N29			
F34				R29			
H34				R39			
P511				R79			
H511							
R111							

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

SHIFT CHECK SHEET

Operator _____ Shift _____ Date _____

I - OFF-GAS SYSTEM

A. Scrubbers

Scrubber No.	Liquid Level	Type Solution	Scrub. Temp.	Flow Temp.	Pressure Drop
N120	____ %	_____	____ °C	____ °C	____ " H_2O
N220	____ %	_____	____ °C	____ °C	____ " H_2O

Scrubbers Changed _____ (4-12 shift only)

B. Filters

	120S	120P	220S	220P
Pressure Drop, " H_2O	_____	_____	_____	_____

C. General

Cell off-gas _____ " H_2O Off-gas flow _____ cfm

Process cell off-gas _____ " H_2O Off-gas temp. _____ °C

Manipulator cell off-gas _____ " H_2O Off-gas monitor _____ cpm

II - CELL VENTILATION SYSTEM

A. Individual Cell Readings

	10E	10W	11	12	13	14E
(-) Pressure, " H_2O	_____	_____	_____	_____	_____	_____
	14W	**15**	**16**	**18**	**19**	**Process**
(-) Pressure, " H_2O	_____	_____	_____	_____	_____	_____

B. Filters

Supply pressure, (-) " H_2O _____

Pressure drops (house):

West bank _____ " H_2O: Middle bank _____ " H_2O: East bank _____ " H_2O

Pressure between house and pit, (-) " H_2O _____

Pressure drops (pit):

CWS _____ " H_2O: Nevaclogs _____ " H_2O

C. General

Pit sump operating _____ Duct sump operating _____

Cell ventilation monitor _____ cpm

III - ^{90}Sr SOLUTION STORAGE

Tank	L.L.	Temp, °C	Tank	L.L.	Temp, °C
P23	____ %	_____	P34	____ %	_____
F23	____ %	_____	F34	____ %	_____
H23	____ %	_____	H34	____ %	_____

(Immediately report to supervision if any tank temperature is >80°C; if any L.L. is >70%; or if any L.L. is <30% and override switch is not on).

IV - GENERAL

A. Sump liquid levels: Cell 11W _____ " H_2O;

B. Cell 10 Storage Wells:

Temp., °C (30°C min, 70°C max) _____

Water level, inches water _____

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

DAILY CHECK SHEET

Operator_____ Date_____

A. Storage and Service Cell Sumps

	Cell 20	Cell 21	Cell 22	Cell 23	Cell 24	Cell 25	Cell 26	W19,W20 Pit
Liquid Level	____	____	____	____	____	____	____	____
Jetted to Waste	____	____	____	____	____	____	____	____

B. Demineralizer:
Meter reading_____, grains/gal_____

C. MH209 (Process Waste)
Integrator reading_____; Previous reading_____ 24 hr total gal_____

D. Chilled Water Units
Unit #1: Inlet temp._____°C; outlet temp._____°C
Unit #2: Inlet temp._____°C; outlet temp._____°C
Pump #1 operating_____; pressure_____
Pump #2 operating_____; pressure_____

E. Yellow Dumpster - West
Not full_____ Full_____ HP Tag_____

F. Yellow Dumpster - East
Not full_____ Full_____ HP Tag_____

G. Building Ventilation Fan and Filters
Motor operating_____; Louvers operating_____
PDRF_____" H_2O; PDAF_____" H_2O; PDI_____" H_2O
Building ventilation_____" H_2O

H. Building Light Checks (record location)
Inside_____
Outside_____
Safety showers_____

I. Remarks
_____
_____
_____
_____
_____
_____
_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

DAILY CHEMICAL INVENTORY

Operator _____ Date _____

MAKEUP TANKS

Tank	Chemical	Gallons	Remarks	Tank	Chemical	Gallons	Remarks
M1134	60% HNO_3			M2			
M1234	8M NH_4NO_3			M3			
Truck	8M NH_4NO_3			M4			
M131				M5			
M231				M532			
M331				M732			
M431				M632	60% HNO_3		

PROCESS CHEMICALS

Chemical	Container	Quantity	Max.	Min.	Remarks
60% HNO_3	Drum		30	15	
70% HNO_3	Case		10	5	
NaOH (flake)	100 lb drum		4	2	
NaOH (C.P. pellets)	5 lb bottle		10	5	
NH_4OH	Case		6	3	
HCl	Case		4	2	
Alum	100 lb bag		100	50	
NH_4CO_3 (C.P.)	400 lb drum		2	0.5	
AMSCO	55 gal drum		4	2	
D2EHPA	55 gal drum		4	2	
Argon	Cylinder		-	4	
TiO_2	1 lb bottle		10	5	
Oxalic acid	300 lb drum		2	1	

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

WEEKLY (FRIDAY) CHECK LIST

Operator_____ Date_____

RECORD LOCATION OF INOPERATIVE ITEMS

A. Emergency lanterns_____

B. Emergency siren check_____

C. Entrance door locks_____

D. Entrance door crash bars_____

E. Assault mask boxes filled_____

F. Emergency cabinets stocked_____

G. Airlock interlock systems_____

H. Containment system (check with supervision before testing)

Initial building pressure_____ "H_2O

Final building pressure_____ "H_2O

Time required_____ sec.

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

WEEKLY (FRIDAY) GENERAL SUPPLIES INVENTORY

Operator _____ Date _____

Item	Quantity	Max	Min	Remarks
Paper suit, 3 pc		150	50	
Airline suit, 2 pc		50	20	
Airline vest		20	10	
Airline hose, 25'		40	20	
Assault mask		50	20	
Respirator		50	20	
Rubber gloves, pr		300	100	
Cloth gloves, pr		300	100	
Plastic boots, pr		300	100	
Cloth hood		150	50	
Blotter paper, roll		2	1	
Wipes, case		10	2	
Kraft paper, roll		4	2	
Masking tape, roll		100	20	
Plastic bags, roll		4	2	
.004" plastic, roll		4	2	
Turkey bags, box		2	1	
3" Dumpster liner		3	1	
3" Dumpster cover		3	1	
1" Dumpster liner		3	1	
Mop bucket		10	5	
Mop handle		24	12	
Mop head		48	12	
Commode brush		24	12	
B-120 filter		4	2	
B-120 filter can		-	5	
B-220 filter can		-	5	
24" x 24" inlet filters		48	24	
Bucket, 1-gal		100	25	
Bucket, 2-gal		100	25	
Bucket, 5-gal		50	10	
Waste can, 30-gal		25	5	
Versene, 100 lb		2	1	
TURCO-4502, 100 lb		2	1	
IGEPAL, 55-gal drum		2	1	
Liquid soap, case		3	1	
Sashcord, hank		10	5	
Oxalic acid, 300 lb drum		2	1	
Ammonium carbonate, 100 lb drum		2	1	

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

SHUTDOWNS

I. Temporary Shutdown with Full-Time Patrol

A. Processing Shutdown

1. Supervision will coordinate the plant shutdown with logical stopping points in processing operations.

   a. All process cell operations, with the possible exception of the ^{137}Cs crystallizer system, will be shut down.

   b. All manipulator operations, with the possible exception of calorimetry assay operations and furnace operations, will be shut down.

2. In the event that ^{137}Cs crystallizer operations are to be continued, supervision will provide specific instructions to the patrol operator. Also, an adequate supply of alum will be packaged in 50-pound bags for use by the patrol operator.

3. In the event that calorimetry and/or furnace operations in the manipulator cells are to be continued by the patrol operator, supervison will provide specific instructions to the patrol operator.

   a. No operations which require insertion of equipment into the cells will be scheduled.

   b. No operations requiring full-time attention in the manipulator cells will be scheduled.

4. All waste in the manipulator cells will be packaged, with special attention given to combustible materials.

5. Solid radioactive materials in the manipulator cells will be stored or covered and placed in a safe location within the cells.

6. Manipulator arms, except those needed for the operations described in Step I.A.3, will be set in positions which will minimize radiation and heat effects; then they will be locked out.

7. All waste tanks will be emptied.

8. Supervision will determine which process tanks require that

agitators and/or chilled water be left on.

9. A "Shift Tank Inventory" will be done.

B. Equipment and Services Shutdown

1. All cell blocks will be installed and sealed.

2. The yellow Dumpster will be emptied. Any shielded Dumpsters that are in the building will be closed and sealed.

3. The chilled water system will be checked for proper operation. If the chilled water system is not operating properly, the vessel cooling system will be switched to process water.

4. The main power switches to all furnaces (except those needed in Step I.A.3) will be turned off.

5. The cell ventilation Neva-clog filters will be washed. After the filters have been washed, the cell ventilation pressures in all cells will be adjusted to -1 inch water.

6. Both off-gas Neva-clog filters will be washed and the scrubbers changed.

   a. If crystallizer operations are to be continued, the standard scrubber solutions will be used.

   b. If no crystallizer operations are to be done during the shutdown, the scrubbers will be changed out but water only (no chemicals) will be added back to the scrubbers.

   c. Normally the B220 filters will be left on stream.

7. The checks shown on the "Weekly Check List" will be made, and any indicated repairs or adjustments will be done.

8. A special effort will be made to assure that all radiation, contamination, and control zones are properly identified. Any unusual zoning situations will be noted in the instructions to the patrol operator.

9. Supervision will arrange for Health Physics to check all radiation monitoring instruments.

10. A check will be made to assure that all agitators (except as noted in Step A.8) and centrifuges are off.

C. Full-Time Patrol Operator Duties

1. Supervision will enter specific instructions concerning ^{137}Cs crystallizer processing and/or manipulator cell work (calorimetry or furnace operations only) in the "Patrol Operator's Log." In addition, supervision will provide the following items to the patrol operator:

   a. Current "Shift Tank Inventory."

   b. "Shutdown Check List."

   c. Name and telephone number of the FPDL supervisor who is on call during the shutdown.

2. The patrol operator will answer all alarms and proceed as instructed in the "Procedure for Patrol Operators."

3. Once each hour the patrol operator will call Guard Headquarters and report his status.

4. The operator will make the checks listed on the "Shift Tank Inventory" and the "Daily Check Sheet" once per shift.

   NOTE: Specific items may not require checking; these will be designated by supervision during shutdown operations.

5. The "Patrol Operator's Log" will be used to record any alarms and required actions in addition to being used to keep a record of activities during the shift.

6. The operator will perform any duties specified by supervision (see Step C.1 above).

7. In case of unusual occurrences not covered by instructions, the operator will contact the Laboratory Shift Supervisor.

8. The patrol operator will remain at the FPDL at all times except in emergencies unless relieved by FPDL supervision or the Laboratory Shift Supervisor.

   NOTE: There are occasions when checks are required on equipment in the Isotopes Area; these will be included in the patrol instructions.

II. Full Shutdown with Part-Time Patrol

A. Processing Shutdown

1. Supervision will coordinate the shutdown with logical stopping points in processing operations; all operations will be shut down.

2. All waste in the manipulator cells will be packaged, with special attention given to combustible materials.

3. Solid radioactive materials in the manipulator cells will be stored or covered and placed in a safe location within the cells.

4. All manipulator arms will be set in positions that will minimize radiation and heat effects; then they will be locked out.

5. All waste tanks will be emptied.

6. Supervision will determine which process tanks require that agitators and/or chilled water be left on.

7. A "Shift Tank Inventory" will be made.

B. Equipment and Services Shutdown

1. All cell blocks will be installed and sealed.

2. The yellow Dumpster will be emptied. Any shielded Dumpsters that are in the building will be closed and sealed.

3. The chilled water system will be checked for proper operation. If the chilled water system is not operating properly, the vessel cooling system will be switched to process water.

4. The main power switches to all furnaces will be turned off.

5. The cell ventilation Neva-clog filters will be washed. After the filters have been washed, the cell ventilation pressures in all cells will be adjusted to -1 inch water.

6. Both off-gas Neva-clog filters will be washed and the scrubber solutions will be changed out, but water only (no chemicals) will be added back to the scrubbers. Normally the B220 filters will be left on stream.

7. The following steam headers will be turned off:

   a. 65-psi header.

   b. 30-psi header.

   c. Cell 18 sampler header.

   d. Tank farm steam header.

8. The checks shown on the "Weekly Check List" will be made, and any indicated repairs or adjustments will be done.

9. A special effort will be made to assure that all radiation, contamination, and control zones are properly identified. Any unusual zoning situations will be noted in the instructions to the patrol operator.

10. Supervision will arrange for Health Physics to check all radiation monitoring instruments.

11. A check will be made to assure that all agitators (except as noted in Step II.A.6 above) are turned off.

12. Red markers will be placed on all alarms that are in alarm condition.

C. General Items

1. All lunchroom equipment except the refrigerator will be turned off.

2. The janitor foreman will be notified of the shutdown.

3. Supervision will contact the Operations Division, inform them of the shutdown, and arrange for testing the alarm relay from the FPDL to the Operations control panel.

4. Supervision will contact the Laboratory Shift Supervisor and inform him of the shutdown.

D. Part-Time Patrol Operator Duties

1. Under the direction of the Laboratory Shift Supervisor, the patrol operator will patrol the building on an established schedule. The preferred schedule is two times each shift, approximately 2 hours after the start of the shift and 2 hours before the end of the shift. However, the actual schedule will be established for each shutdown.

2. The patrol operator will check the four main areas of the building (1st level north and south, 2nd level north and south) and will take the required data to fill out the "Patrol Operator's Log Sheet."

3. At times it will be necessary for the patrol operator to perform certain procedures. These are described in "Patrol Operator's Duties."

4. The patrol operator will answer all alarms and proceed as indicated in "Patrol Operator's Duties."

III. Startup Procedures

A. Startup After Temporary Shutdown

1. Supervision will review the "Patrol Operator's Log" to determine the status of operations and will direct any necessary action indicated by problems that occurred during the shutdown period.

2. A "Shift Tank Inventory" will be made and the items on the "Shift Check Sheet" and the "Daily Check Sheet" will be checked. Supervision will then determine any adjustments or further checking that may be required before operations are resumed.

3. The off-gas scrubber solutions will be changed out.

B. Startup After Full Shutdown

1. Supervision will review the "Patrol Operator's Log Sheets" that were filled out by the part-time patrol operators during the shutdown. If any prestartup actions are indicated because of problems which occurred during the shutdown period, supervision will so direct.

2. The items shown on the "Shift Check Sheet," the "Daily Check Sheet," and the "Weekly Check Sheet" will be checked, and a "Shift Tank Inventory" will be made. Any necessary actions indicated by the results of these checks will be directed by supervision.

3. The following steam headers will be turned on:

   a. 65-psi header.

   b. 30-psi header.

   c. Cell 18 sampler header.

   d. Tank farm sampler header.

4. Lunchroom equipment will be turned on.

5. A check will be made of all manipulator boots to assure that they are in good condition; then the manipulator arms will be unlocked.

6. Supervision will arrange for Health Physics to check all radiation monitoring instruments.

7. The off-gas scrubber solutions will be changed out.

8. The red markers will be removed from the panelboard alarm annunciators.

9. Supervision will contact the Laboratory Shift Supervisor, the Operations Division, and the janitor foreman to inform them of the startup.

FISSION PRODUCTS DEVELOPMENT LABORATORY

SHUTDOWN CHECK LIST

Date_____ Type shutdown: Temporary_____ Full_____

Processing Shutdown	Supr.
Process operations shutdown*	_____
Alum packages prepared (if required)	_____
Manipulator cell waste status checked	_____
Radioactive solids storage checked	_____
Manipulators positioned and locked out**	_____
Waste tanks emptied	_____
Process tank status	
Agitation on_____	
Chilled water on_____	
Shift tank inventory complete	_____

Equipment and Services Shutdown	
Cell blocks sealed	_____
Dumpsters secured	_____
Chilled water system checked	_____
All furnace switches off***	_____
Cell ventilation Nevaclogs washed	_____
Hot-off-gas Nevaclogs washed	_____
Scrubber solutions changed	_____
Weekly check list complete	_____
Zoning check complete	_____
H.P. instruments checked	_____
Agitators checked	_____
Steam headers off	_____
Lunchroom equipment off	_____
Red markers placed on alarms	_____

Remarks

*For temporary shutdown, describe process operations_____

**For temporary shutdown, list manipulators to be used_____

***For temporary shutdown, list furnaces to be used_____

Miscellaneous_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

PATROL OPERATOR'S DUTIES

I. GENERAL

A. Patrol Schedule for Part-Time Patrol

The Laboratory Shift Supervisor will establish the schedule for patrol. Normally the patrol will be made twice each shift.

B. Patrol Checks and Records

The patrol operator will check the four main areas of the building (1st level north and south, 2nd level north and south). At each patrol check, the operator will fill out a "Patrol Operator's Log Sheet." Any actions indicated by the results of the checks will be taken immediately. Blank log sheets are kept on the clipboard, which is also used for completed sheets. This clipboard will be replaced in the shift supervisor's office after the patrol check is complete.

II. Alarm Procedures

A. Process Emergency Panel

The Process Emergency Panel is located in the 2nd level north area of the building. Alarms on this panel will sound the air horn signal. Patrol operators will proceed as indicated in the following procedural list. (LSS = Laboratory Shift Supervisor; HP = Health Physics representative; FPDL Supr = FPDL on-call supervisor.)

Alarm		Procedure
HI RADIATION CONDENSATE DRAIN	Operator:	Contact LSS and HP.
	HP:	Survey service tunnel drain.
	LSS:	Contact FPDL Supr if radioactivity is detected in drain.
HI RADIATION CHILLED WATER	Operator:	Contact LSS and HP.
	HP:	Survey chilled water unit.
	LSS:	If radioactivity is detected in unit, zone the area and contact FPDL Supr.
HI RADIATION SERVICE TUNNEL	Operator:	Contact LSS and HP.
	HP:	Survey service tunnel.
	LSS:	Contact FPDL Supr and transmit survey results.

Alarm	Procedure	
HI RADIATION CELL VENTILATION	Operator:	Contact stack operator to advance tape. If high radiation continues, contact LSS and FPDL Supr.
LOW PRESSURE INSTRUMENT AIR	Operator:	Acknowledge and record in log.
PROCESS CELL VENTILATION HI PRESSURE	Operator:	Contact stack operator and check FPDL cell ventilation filters. Contact FPDL Supr and give him filter pressure drop results.
HI PRESS OFF-GAS MANIP & PROC CELLS	Operator - Full-Time:	Check off-gas filters. If Neva-clog is plugged, switch filter banks and wash Neva-clogs; then switch back to original filters. If problem is not in Neva-clogs, contact FPDL Supr.
	Part-Time:	Check FPDL off-gas filters and then contact FPDL Supr.
BUILDING CONTAINMENT ALARM	Operator:	Contact LSS and HP.
	HP:	Check hi-vols and CAM's. If radioactivity is detected, zone the building.
	LSS:	If radioacitivity is detected, contact FPDL Supr. If no radioactivity is present, reset containment and start fan. If system will not reset, contact FPDL Supr.
R18 HIGH LEVEL	Operator:	Contact FPDL Supr.
MANIPULATOR CELL VENTILATION HI PRESSURE	Operator:	Contact stack operator and check FPDL cell ventilation filters. Contact FPDL Supr and give him filter pressure drop results.
LOW PRESSURE CHILLED WATER	Operator:	Contact FPDL Supr.
LOW PRESS BUILDING STEAM	Operator:	Acknowledge and record in log.

Alarm	Procedure	
BAY AREA EMERGENCY	Operator:	Check for personnel in bay area.
CELL 3 & 4 TEMP OR LEVEL	Operator:	Check liquid levels and temperaturès on tanks P23, H23, F23, P34, H34, and F34; then contact FPDL Supr.

B. Radiation Alarms

Radiation monitors will alarm locally and on the central panel in the office on the 2nd level south. After acknowledging the alarm, the patrol operator will contact Health Physics. Health Physics will survey the area indicated and will check the instrument.

1. If a radiation condition exists, Health Physics will establish the proper zones and contact the Laboratory Shift Supervisor, who will contact the FPDL on-call supervisor.

2. If the trouble is with the instrument, the operator will record this in the log. Health Physics will attach an out-of-service tag if necessary.

C. High Solution Level in Off-Gas Scrubbers (N-120 or N-220, located on 2nd level SE)

1. If N-120 liquid level indicator alarms or if the liquid level is above 60% at one of the routine checks, the patrol operator will proceed as follows:

   a. Open valve switch HX-22033.

   b. Open jet switch HX-J120, and jet solution from N-120 until the N-120 liquid level is 30%; then close jet switch HX-J120.

   c. Close valve switch HX-22033.

2. If N-220 liquid level indicator alarms or if the liquid level is above 60% at one of the routine checks, the patrol operator will proceed as follows:

   a. Open valve switch HX-22035.

   b. Open jet switch HX-J220 and jet solution from N-220 until the N-220 liquid level is 30%; then close jet switch HX-J220.

   c. Close valve switch HX-22035.

D. High Solution Level in Condensate Tank (H209, located on 2nd level NW)

If the H209 liquid level indicator alarms or if the liquid level is above 70% at one of the routine checks, the patrol operator will proceed as follows:

1. Turn on pump ES-CT.

2. Pump solution from H209 to M.H.209 until the H209 liquid level reaches 20%.

3. Turn off pump ES-CT.

E. Panelboard Annunciator Alarms

Panelboard annunciators that are in an alarm condition at the time of the shutdown will be marked with red magnetic buttons. If any other annunciators alarm, the patrol operator will contact the FPDL on-call supervisor.

F. Washing of Neva-Clog Filters in Off-Gas System

It will sometimes be necessary to wash the Neva-clog filters in the off-gas system because of reduced off-gas supply. The washing will be done as needed by the full-time patrol operator. When part-time patrol is being done, the patrol operator will contact FPDL supervision before washing the filters. The procedure for washing the B120 filters is as follows:

1. Open the upstream and downstream manual valves on the B220 off-gas filters. These valves are located on top of Cell 20.

2. Close the upstream and downstream manual valves on the B120 off-gas filters. These valves are located on top of Cell 20.

3. At the first level northeast side of the building (next to Cell 20), connect the Chicago-coupling fitted jumper line to the line labeled 120W. Open the two valves on this line and allow the water to flow for 2 minutes. Then close both valves and disconnect the Chicago coupling.

4. Repeat Step 3 but connect the jumper to the line labeled 120E.

5. Allow the system to drain for 15 minutes.

6. Open the upstream and downstream manual valves on the B120 off-gas filters.

7. Close the upstream and downstream manual valves on the B220 off-gas filters.

FISSION PRODUCTS DEVELOPMENT LABORATORY

PATROL OPERATOR'S LOG SHEET

Date_____ Shift_____ Time_____ Operator_____

Item	Location	Limit*	Value
N-120 liq. lev.	2nd lev. SE	60% max.	
N-220 liq. lev.	2nd lev. SE	60% max.	
PD 120S	2nd lev. SE	3 inch max.	
P-PCOG	2nd lev. SE	30%-70%	
P-MCOG	2nd lev. SE	30%-70%	
3517 Off-gas Pressure	2nd lev. NE	25 inch min.	
H209 liq. lev..	2nd lev. NW	70% max.	
S324 liq. lev.	2nd lev. NW	70% max.	
S223 liq. lev.	2nd lev. NW	70% max.	
S523 liq. lev.	2nd lev. NW	70% max.	
P23 liq. lev.	2nd lev. NW	70% max.	
F23 liq. lev.	2nd lev. NW	70% max.	
H23 liq. lev.	2nd lev. NW	70% max.	
P34 liq. lev.	2nd lev. NW	70% max.	
F34 liq. lev.	2nd lev. NW	70% max.	
H34 liq. lev.	2nd lev. NW	70% max.	
Process Cell Pressure	1st lev. NW	(-) 0.5 inch min.	
Manip. Cell Pressure	1st lev. SW	(-) 0.5 inch min.	
Storage well Temp.	1st lev. SW	40°C max.	

*If either N-120 or N-220 liq. lev. is above 60%, jet solution to waste to 30% liq. lev. - see "Procedure for Patrol Operators." If any other listed limit is exceeded, contact FPDL supervision.

Remarks: _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERAMIC FILTER BUILDUP

NOTE: This procedure applies to filter assemblies in either the Cell 11 (P511) system or the Cell 14E (P114) system. Both systems are identical in construction and use the same size filter thimbles, gaskets, and lines.

A. Preassembly Check (Outside the Cell)

1. Check each filter thimble for vertical alignment with a square. If a filter thimble shows greater than 1/8-inch deviation from end to end while resting in a vertical position on a flat plate, repair it or reject it. The sealing rims of the thimbles can be repaired by rotating them on a flat metal surface until the rim is smooth.

2. After the thimble is repaired, check that the thimble height is 11 3/4 inches, ± 1/16 inch.

3. Check the filter for cracks and for cleanliness.

4. Bag the filters and the proper gaskets and lines and insert them into the cell according to the "Airlock Operation."

5. Replace the filter gaskets and pads before each run with the following (OD = outside diameter; ID = inside diameter):

   a. Filter shell cover gasket: 7 3/4-inch-OD Tygon O-ring made from 1/4-inch-OD by 1/8-inch-ID Tygon tubing.

   b. Filter thimble gasket: two flat Tygon disks with trefoil cutouts (see Dwg. A-RD-1789).

   c. Filter thimble pads: 6-inch-OD by 2-inch-ID O-ring made from 1/8-inch-thick flat Tygon. These pads have several holes punched in them.

6. If so instructed by supervision, replace the following:

   a. P511 adapter gasket: 8 7/8-inch-OD Tygon O-ring made from 1/4-inch-OD by 1/8-inch-ID Tygon tubing.

   b. Tygon lines (1/2-inch-ID by 1/8-inch wall tubing) of the following lengths:

From precipitator to primary filter:	14 inches
From primary to secondary:	20 inches
From secondary to safety bottle:	24 inches
From safety bottle to receiver:	24 inches

B. Assembly (In-Cell)

1. Check the gasket seal surfaces. Remove any appreciable residue from the surfaces by wiping. (CAUTION: Do not scrape the seal surfaces with sharp metal objects; scratches will affect the seal.) Check the bottom and sides of the filter shell and remove any residue.

2. Depending on the filter height (check with supervision), install one or two filter thimble pads in the bottom of the filter shell. Be sure that the pads lie flat.

3. Using the filter lifting tool, install the filter thimble in the center of the shell. Use a mirror to check for center alignment and vertical height. The filter thimble should project approximately 1/16 inch above the sealing surface of the filter shell.

4. Install _____ filter thimble gaskets on the filter shell cover (check with supervision for number of gaskets).

5. Install the Tygon O-ring gasket (filter shell cover gasket) on the seal surface of the filter shell. Check for alignment.

6. Carefully place the filter shell cover on the filter shell.

7. Engage the V-bank clamp around the flange and tighten securely. Use extra leverage on the cross handle for final seal.

8. Repeat the operation for the secondary filter.

9. Connect the filters to the process connections as described in the appropriate procedure.

10. Check with supervision for the filter test procedure to be used.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CALORIMETER OPERATION

NOTE: This procedure applies to both the ^{90}Sr calorimeter and the fast ^{137}Cs calorimeter. However, since the fast ^{137}Cs calorimeter is the larger it is routinely used for measuring either ^{137}Cs or ^{90}Sr samples. The ^{90}Sr calorimeter can be used for only ^{90}Sr samples and is not generally used.

A. Operational Procedure

1. Check that the following equipment is in Cell 14-W and is in good operation condition.

   a. Calorimeter base.

   b. Calorimeter cover.

   c. Dewar flask fitted to calorimeter base.

   d. Cooling water lines and connections.

2. Connect the cooling water inlet and outlet lines to the calorimeter base. Check with supervision to determine if chilled water or recirculated water is to be used, and set the valves. If recirculated water is to be used, see that the recirculation pump is operating and check the level of the water tank (it should be at least half full).

3. Set the water flow to the calorimeter base. Set rotometer No. 1 at _____ and rotometer No. 2 at _____.

4. Place the Dewar flask on the cell floor next to the calorimeter base. Remove the calorimeter cover and set it on the floor.

5. See that the calorimeter cup is empty.

   NOTE: Two sets of calorimeter cups are used -- one set for ^{137}Cs and one set for ^{90}Sr. The cups are identical except that the ^{90}Sr cups are marked "^{90}Sr" on the sides and top. The cups normally stay in specific cells and care must be taken to ensure that they do not get mixed up.

6. Do <u>not</u> transfer a ^{90}Sr cup to a ^{137}Cs cell or vice versa unless it is going directly to the calorimeter.

7. Tare the calorimeter cup.

8. Determine from supervison the designation of batch to be sampled and check the approximate sample weight to be measured. Put the powder into the cup and reweigh the cup plus powder. Calculate the sample weight and record it on the appropriate data sheet.

9. Screw the calorimeter cup lid onto the cup. Do not force the lid on; just finger-tighten it. Use the calorimeter cup lifting tool for this operation.

10. Transfer the loaded calorimeter cup to Cell 14-W, using appropriate transfer procedure (see "Airlock Operation").

11. Set the calorimeter cup into the calorimeter. Set the cover on the calorimeter.

12. Position the Dewar flask over the calorimeter so that the rim of the flask lies in the channel in the calorimeter base.

13. Check the system once every 30 minutes and record the following data on the data sheet:

    a. Calorimeter temperature (in millivolts).

    b. Calorimeter base temperature (in $^{\circ}C$).

    c. Rotometer No. 1 flow (adjust to _____ as necessary).

    d. Rotometer No. 2 flow (adjust to _____ as necessary).

14. Continue to operate until the calorimeter temperature reading has been constant ± 0.02 millivolt for 2 hours (five consecutive readings) and the calorimeter base temperature has been constant $\pm 1^{\circ}C$ over the same period.

15. Record the final calorimeter temperature reading on the appropriate data sheet.

16. Remove the Dewar flask from the calorimeter. Remove the calorimeter cover.

17. Lift the calorimeter cup out of the calorimeter and transfer it back to the cell from which it came. Remove the powder from the cup and combine it with the remainder of the batch. Check with supervision for storage instructions.

    NOTE: Supervision will advise whether other measurements are to be made or the calorimeter is to be shut down. When a series of measurements of the same type are being made, a sample should be prepared in advance so that it will be ready to insert into the calorimeter as soon as

NOTE (Continued): the previous sample has been removed. When this is done, the calorimeter base retains its heat and constant readings are reached quicker. Duplicate calorimeter cups are provided for this purpose.

B. Calculations of Specific Activity of Powder

1. Definitions:

   a. The calorimeter temperature (T) is expressed in millivolts. It is a measure of the heat output of the sample. The number used in the calculation is the one recorded in Step A.15.

   b. The sample weight (W) is expressed in grams and is determined in Step A.8.

   c. The calorimeter factor (F) is expressed in watts per millivolt. This factor is determined for each calorimeter by calibration against a known heat source. Different factors are needed for ^{90}Sr and ^{137}Cs. They are calculated as of the time a new calorimeter is installed and are posted (see opposite page).

   d. The conversion factor (C) is expressed in watts per curie. This factor differs for ^{90}Sr and ^{137}Cs, and the individual factors will vary according to the amounts of ^{89}Sr and ^{134}Cs in the samples. (Current values are posted on the "Special Instructions" sheet opposite.)

2. Calculation:

   a. Calculate the specific activity of the powder (curies per gram) according to the formula given below and record it on the data sheet:

$$\frac{T\ (\text{millivolts}) \times F\ (\text{watts/millivolt})}{W\ (\text{grams}) \times C\ (\text{watts/curie})} = \underline{\qquad\qquad}\ \text{curies/gram.}$$

C. Calorimeter Calibration

1. Electrical Calibration

Calibration of the calorimeter with an electric heater is done by I and C personnel as scheduled by the FPDL supervisor. When an electrical calibration is to be made:

   a. Connect the test heater to the outlet in Cell 14-W.

   b. Insert the heater cup into the calorimeter; then place the test heater into the cup.

c. Position the electrical lead wire in the groove in the calorimeter base and place the Dewar on the calorimeter.

d. Turn on water to the calorimeter base and set rotometers No. 1 at _____ and No. 2 at _____.

e. When I and C personnel have completed the calibration, turn off cooling water. Then remove the test heater and heater cup from the calorimeter.

2. ^{90}Sr Source Check

Supervision will direct the ^{90}Sr source check of the calorimeter (usually daily during ^{90}Sr processing campaigns); the operator's duties are as follows:

a. Perform Steps A.1 through A.4.

b. Remove the designated standard ^{90}Sr source from storage and insert it into the calorimeter (no cup is used).

c. Perform Steps A.11 through A.16.

d. Remove the ^{90}Sr source from the calorimeter and return it to storage.

e. Calculate the heat output measured,

T (millivolts) X F (watts/millivolt) = _____watts,

and compare it to the recorded value of the standard. If the value varies as much as 2%, contact supervision.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CALORIMETER RECORD

Date _____

Material _____ Batch Number _____

Sample Weight _____ Radiochemical Composition _____

Calorimeter Number _____ Date Last Calibration _____

Calorimeter Factor _____ w/m.v. Conversion Factor _____ w/c

Time	Date	Cal. Temp., m.v.	Base Temp. °C	Cell Temp. °C	Roto. No. 1, %	Roto. No. 2, %	Initials and Remarks

FISSION PRODUCTS DEVELOPMENT LABORATORY

AIRLOCK OPERATION

1. Check that all airlock doors are closed. If any of the permanent airlock doors are not operable, check that a movable airlock baffle is in place.

2. Check that no other transfers will be necessary during the time required to make this transfer.

3. Each time a dolly is moved between cells:

   a. Check that all items on the dolly are positioned so that they will not touch the sides or top of the airlock.

   b. Open the airlock door nearest the dolly, slide the dolly into the airlock, then close the door.

   c. Open the other airlock door, withdraw the dolly, then close the airlock door.

4. See that at no time during a transfer are two airlock doors open at the same time.

5. If an airlock door is found to be inoperable, report it to supervision immediately. Also inform any personnel working in other manipulator cells that the airlock is out of service.

6. Move the dolly slowly and as smoothly as possible. This is especially important when transferring liquids in bottles or powders in covered vessels.

7. Always check the transfer track to make sure that it is free from obstructions before making a transfer.

FISSION PRODUCTS DEVELOPMENT LABORATORY

BLENDER OPERATION

A. Equipment Setup

The blenders used at the FPDL are standard Waring blenders with a stainless steel cup and cover. Manipulator channel grips are installed on the cup and cover. The blender base may be either porcelain or stainless steel.

1. Before a blender is inserted into a cell, the channel grips should be checked for proper installation and for proper operation.

2. Other equipment used during blending operations is standard manipulator cell equipment. Blending procedures are issued individually, with each procedure giving the number of blends, the weights of the various fractions to be blended, the batch numbers, and the number of vessels needed for handling. Vessels will be identified as "storage" vessels or "receiver" vessels. The storage vessels will be those containing material to be blended, and the receiver vessels will be those used to accept blended material. Since it is impractical to mark the various vessels for each blending operation, identification of vessels depends on positioning in the cell. The positions of the vessels are recorded on notes on the cell records.

3. Before the blending operation is started, all necessary equipment must be in place and the positions of the various vessels recorded.

B. Operation

1. Weigh the proper amounts of the powders to be blended -- according to the blending procedure -- and add the powders to the blender cup.

2. Set the cover on the blender cup.

3. Set the blender on the base and engage the drive.

4. Set speed control to "low" and turn on the blender. Blend for 1 minute and then turn off blender.

5. Without removing the cover, lift the blender off the base, tilt to ~60° from vertical, and tap it lightly on the floor.

6. Repeat Steps 3, 4, and 5 for a total of four times.

7. Replace the blender on the base, let it stand for 5 minutes, then tap it lightly on the sides with a spatula. Let the blender stand for an additional 5 minutes.

8. Carefully remove the cover. Transfer the powder from the blender to the appropriate receiver vessel (see blending procedure).

NOTE: A thin film of powder will adhere to the sides of the blender cup. It is not necessary to remove this powder until after the last blend has been made.

FISSION PRODUCTS DEVELOPMENT LABORATORY

ULTRASONIC CLEANER OPERATION

1. Check with supervision for identification of items to be cleaned. Clean the items as well as possible in the high-level cells before transferring them to Cell 15.

2. See that special hooks, racks, or other tools needed for handling the items to be cleaned are in Cell 15.

3. Check with supervision for the sequence of cleaning solutions to be used and make up solutions as instructed.

4. Check with supervision that no entries to Cell 15 are scheduled.

5. Add cleaning solution to the ultrasonic tanks to the point at which all transducers are covered.

6. NOTE: Do not operate the ultrasonic system unless both tanks are full of solution.

7. Transfer the items to be cleaned into Cell 15 and place them in the baskets of the ultrasonic tanks.

8. Check that all switches are off and all dials are in the "1" or "0" position.

9. Turn on main switch and set timer to specified time.

10. NOTE: Do not exceed 50 on the timer. Never operate the ultrasonic system on "hold."

11. Wait 2 minutes and then turn on Plate Power.

12. Adjust Line Voltage to 100%.

13. Turn Loading dial to adjust to approximately 450 milliamps.

14. NOTE: Do not exceed 475 milliamps.

15. Use Tuning dial to adjust to 450 milliamps.

16. Operate for time specified.

17. NOTE: On instructions by supervision, items may be removed while the ultrasonic system is operating. When removing items, rinse them with a fine stream of water while they are being drawn through the surface of the cleaning solution.

18. Set Tuning dial to 1.

19. Set Loading dial to 1.

20. Set Line Voltage to 1.

21. Turn off Plate Power.

22. Turn off main switch.

23. Drain both tanks by opening the manual drain valves. When tanks are empty, close the drain valves.

24. Refill the tanks with the next cleaning solution as instructed by supervision; then repeat Steps 8 through 24 for the number of cycles specified.

25. After items have been cleaned, remove them from the ultrasonic tank and handle as instructed by supervision.

26. Always rinse ultrasonic tanks once, then refill with water before shutting down.

FISSION PRODUCTS DEVELOPMENT LABORATORY

HIGH-LEVEL TRANSFERS

NOTE: This procedure applies specifically to the transfer of solid product materials to the Source Development Laboratory (Building 3029). Supervision will designate the materials to be transferred. For other high-level transfers, special loading instructions will be provided by supervision.

A. Preliminary Checks

1. Position the cans in the appropriate location as instructed.

   a. ^{137}Cs products will normally be in Cell 14W.

   b. ^{90}Sr products will normally be in Cell 12.

2. Check that the proper transfer container and handling tools are available in the cell containing the products.

3. Check that the transfer cask is clean and ready to be loaded.

4. Check that any special cask inserts (baskets, chill blocks, etc.) are ready for insertion into Cell 15.

B. Cell 15

1. Wrap the transfer cask in clean plastic. Leave the cask plug unwrapped so that it can be removed. Remove nuts and locking devices from the cask plug.

2. Remove the Cell 15 top block.

3. Insert the transfer cask into Cell 15 and center it.

4. Insert any special cask inserts or tools required in Cell 15,

5. Replace the Cell 15 top block.

6. Remove the small center plug from the Cell 15 top block and insert the through-block choker.

7. Attach the choker to the transfer cask plug and lift the plug about 3 feet.

8. Position the cask inserts, baskets, or other special handling devices as instructed by supervision.

C. Loading Operation

1. Check with supervison for specific loading instructions.

2. Check that operations in Cell 14W are shut down temporarily. (In case the material to be loaded is in any other cell than 14W, check that operations in the intervening cells are also shut down.)

3. Using standard airlock procedures, transfer the shipping cans to Cell 15 and insert them into the transfer cask as instructed by supervision.

4. When all cans have been loaded into the transfer cask, lower the plug onto the cask.

D. Removal of Transfer Cask

1. Check that the Cell 14-15 airlocks are closed.

2. Contact supervision for issuance of Radiation Work Permit.

3. Under Radiation Work Permit conditions, remove the Cell 15 top block.

4. Raise the transfer cask until the top of the cask is even with the top of Cell 15. Remove the plastic wrapping and let it drop back into Cell 15.

5. Survey and smear the cask. Perform preliminary cleaning of cask while it is over Cell 15; then set the cask on paper in the crane bay.

6. Attach nuts and locking devices to the cask.

7. Decontaminate the cask to shipping tolerance. Check with supervision for wrapping instructions -- casks with high heat loads cannot be wrapped in plastic.

E. Transfer to Fission Products Encapsulation Facility

1. Supervision will fill out the Radioactive Materials Transfer tag; Health Physics will perform the required surveys.

2. Supervision will contact the Isotopes Division Radiation Control Officer for approval to make the transfer and also contact Source Development Laboratory to schedule the transfer.

3. Casks that can be handled by the large truck will be placed on the truck and the tie-down chains secured.

4. Two men, a driver and an observer, will make the transfer. The observer will carry a cutie pie and both men will wear PRM's.

5. The truck will be driven directly to Building 3029 at a maximum speed of 15 mph.

6. For casks too large for the large truck, supervision will arrange for transfer by P&E personnel.

FISSION PRODUCTS DEVELOPMENT LABORATORY

MANIPULATOR REMOVAL

A. Preparation

NOTE: Supervision will schedule shutdown of the cell and determine when the manipulator arm is to be pulled.

1. Cover the floor in front of the cell with blotter paper.

2. Cover adjacent panelboards, cabinets, and the cell face with brown wrapping paper.

3. Position CAM near the operating area.

4. Establish zone and control areas (see "Zoning Procedures").

5. Provide the following protective clothing and equipment:

   a. Plastic suits (3).

   b. Vests (3).

   c. Air lines (connected to air supply).

   d. Rubber gloves.

   e. Shoe covers.

   f. Assault masks.

   g. Dosimeters (zeroed).

   h. Cutie pie (10 r/hr), LongTom (20 r/hr), emergency cutie pie, and GM survey meter.

   NOTE: Supervision will place "Entrance Requirement" signs at all entrances to the manipulator area and will specify gas mask, "C" clothing, and shoe covers.

   In the case of Cells 18 and 19, the 31 and 32 makeup areas will become the mask area and all doors leading to these areas will be marked as above.

6. Turn air conditioner off. Controls for the manipulator area units are located on the southeast mezzanine.

B. Removal of Manipulator Arm

Removal of the manipulator arm will be done by members of P and E Division. The operation will have continuous Health Physics coverage. All personnel inside the roped-off area will be in plastic suits. Others in the manipulator area will wear respiratory protection.

C. Removal of Arm from Manipulator Area

If it is necessary for the arm to be taken to the manipulator shop for repairs, Health Physics will determine radiation and contamination levels. Then the operator and P and E personnel will proceed as follows:

1. If the arm is clean enough to go to the shop, the operator will rewrap the slave end in plastic. P and E personnel will remove the arm from the operating area to the manipulator truck (Health Physics tags are required).

2. If the arm is too hot to go to the shop, the operator will rewrap the slave end in plastic and the arm will be taken to Cell 15 via south airlock, White Oak Avenue, 3rd Street, and west airlock. At this point the operator will proceed as follows:

   a. Using the crane, lift the manipulator from the truck and secure it to the manipulator rack.

   b. Hook the crane to the manipulator rack, lift the rack and manipulator, and place them in Cell 15.

   c. Disconnect from the rack and replace the Cell 15 blocks.

   d. Enter Cell 15 (airline suit) and remove the plastic from the SLAVE end of the manipulator. DO NOT REMOVE PLASTIC BAG FROM MASTER END.

   e. Clean slave end to a low enough tolerance to allow arm to be removed from the cell.

D. Cleanup of Manipulator Area

1. After manipulator maintenance is completed, Health Physics checks all personnel for contamination.

2. Health Physics checks the area outside the roped-off area and then the operators clean it.

3. Operators carefully remove paper inside roped-off area; after Health Physics checks the area, the operators clean it.

   NOTE: Supervision will see that "Entrance Requirement" signs are removed.

FISSION PRODUCTS DEVELOPMENT LABORATORY

MANIPULATOR CELL WINDOW CLEANING

A. Safety Requirements

1. Area 33: Fenced off and "No Smoking" signs posted. Hi-Vols and fans turned on. Off-gas to makeup tanks open. Fire extinguishers available.

2. Manipulator Area: Roped off from Cell 27 to Cell 14 and south airlock. "No Smoking" signs posted. Fire extinguishers available.

3. Transfers of Toluene and Alcohol: Will be handled in a closed system vented to off-gas or cell ventilation.

B. Chemical Makeup

1. Pump carboy of acetic acid in a stainless steel tank which has been rinsed with distilled water and dried.

2. Rinse and dry a stainless steel tank. Add 20 gallons of isopropyl alcohol and 20 gallons of "T" thinner (toluene). Use filter stick to clean solution going into tank.

3. Mix thoroughly and leave agitator running.

C. Operations

1. Fill line from solvent tank to cell window.

2. Disconnect N_2 purge and tie in solvent addition line to window long leg. Vent window to Cell 15.

3. Fill window with solvent.

4. Disconnect solvent addition and tie long leg to 55-gallon sealed drum, which is vented to Cell 15.

5. Apply N_2 pressure (2 psi) to vent side of window and empty solvent from window.

6. Repeat above steps if visual inspection indicates more cleaning is necessary.

7. Disconnect solvent lines and tie in acetic line from acid tank.

8. Fill window with acetic acid.

9. Disconnect acetic addition line and tie dip leg to rubber-stoppered acetic acid carboy. Vent carboy to Cell 15.

10. Apply N_2 pressure (2 psi) to vent side of window and empty acetic from window.

11. Repeat Steps 1 through 6 three times.

12. Flush window with new mineral oil.

13. Fill window with mineral oil and leave in overnight. Drain the following morning.

14. Fill window with new mineral oil and hold under an N_2 blanket.

FISSION PRODUCTS DEVELOPMENT LABORATORY

SAMPLE TRANSFERS

A. Sampling from Cell 18 and Cell 19

1. When sampling is called for in a procedure or is requested by supervision, check the sample code and label a sample bottle. Insert the sample bottle into the cell through the sample blister.

2. Insert a bottle into the sample jet station, turn on the sample jet, and recirculate the sampler for 5 minutes or as otherwise instructed.

   NOTE: See Sections F and G for special sampling procedures for vessels C17, C27, C38, C48, P45, R111, and R18.

3. Pour the sample into the labeled sample bottle, tighten the cap on the bottle, and rinse the bottle with water.

   a. If an "A" (supernate) sample has been requested, pour 10 milliliters of solution into a centrifuge tube, centrifuge, and decant into the sample bottles.

   b. If a "B" (precipitate) sample has been requested, check with supervision for dissolving instructions.

   c. If solids are noted in a sample, contact supervision. Normally, no sample will be submitted to the Analytical Laboratory if detectable solids are present.

4. Check that the sample carrier is clean and empty.

5. Remove the base plate from the sample carrier by loosening the wing nuts, rotating the carrier, and lifting it out of the base plate.

6. Position the carrier on the sample removal blister.

7. Release the latch on the carrier cap and open the cap to expose the vacuum adapter.

8. Connect the threaded vacuum line to the vacuum adapter on the carrier.

9. Raise the latch on the bottom drawer of the carrier and withdraw the drawer until it stops.

10. Place the sample in the conveyor cup and run it into the sample blister until it stops under the carrier.

11. Close the vent on the vacuum line and turn on the vacuum.

12. Release the pin on the vacuum adapter and lower it until the vacuum cup engages the cap of the sample bottle.

13. Position a cutie pie survey meter on the cupola in such a manner that it may be watched,and raise the bottle into the carrier by raising the vacuum adapter. Survey the carrier for radiation. If carrier reads greater than 100 mr/hr, lower the bottle into the cubicle and repeat the operation, using the uranium sample carrier.

14. Close the bottom drawer on the carrier; check that latch is secure.

15. Lower the vacuum adapter until the bottle rests on the bottom of the carrier.

16. Open the vent on the vacuum line and turn off the vacuum.

17. Raise the vacuum adapter to the "up" position and engage the pin.

18. Disconnect the vacuum line from the vacuum adapter.

19. Close and latch the carrier cap.

20. Lift the carrier and set it on the base plate. Rotate the carrier to engage the slots in the base plate and tighten the wing nuts. Survey the carrier.

21. Cover the carrier with a plastic bag. Check with supervision for tags and sample sheets.

22. Transfer the sample to the 3019 Analytical Lab (see Section E below).

B. Sampling from Tank Farm (S523, S223, or S324)

1. Insert the labeled sample bottle into the tank farm sampling cubicle.

2. Secure the sample bottle on the sample station.

3. Open the block valve from the tank to be sampled. Check that the block valves to the other two tanks are closed.

4. Open the manual steam valve to the sampler.

5. Turn on the sampler steam.

6. Let the sample pull for 5 minutes; then turn off the sampler steam and close the manual steam valve.

7. Close the block valve between the sampler and the tank.

8. Remove the bottle from the sampler and cap the bottle.

9. Set a lead sample shield into the blister.

10. Transfer the sample bottle into the shield.

11. Remove the shielded sample from the blister. Immediately check the blister for contamination and clean if necessary.

12. Tape the sample shield and transfer it into Cell 18 via the Cell 18 blister.

C. Sampling from Manipulator Cells

1. When a sample is to be taken from a manipulator cell, check the Operational Procedure or contact supervision for the sample code and take the sample as directed.

2. Check that the sample carrier is clean and empty. Wrap carrier in plastic.

3. NOTE: When the uranium sample carrier is used, insert it into Cell 15 from the top and set it on the work table.

4. Enter Cell 15 under appropriate Cell Entry Procedure and set the carrier on the Cell 15 transfer tray, or remove Cell 15 top block and position carrier for loading (check with supervision).

5. Close Cell 15.

6. Rinse the sample bottle and transfer the sample to Cell 14.

7. Raise the carrier cap and remove the plug.

8. NOTE: Secure the vacuum adapter to the plug before raising, in order to prevent it from dropping out. Use the rubber band.

9. Raise the Cell 14 and Cell 15 airlock door and transfer the sample to Cell 15. Close the airlock.

10. Using the Cell 15 vacuum cup, transfer the sample to the carrier.

11. Insert the carrier plug and close the cap.

12. Enter Cell 15 under Cell Entry Procedure. Survey the carrier and smear for contamination. Wrap the carrier in plastic and attach tags. Check with supervision for tags and request sheets.

13. Transfer the sample to the 3019 Analytical Lab (see Section E below).

D. Sampling from Off-Gas Scrubbers

The off-gas scrubbers are sampled only on request by supervision.

1. Open the sample cubicle door and survey the opening for radiation.

2. Using laboratory tongs, insert a sample bottle onto the sample jet station. Close the door.

3. Turn on the air supply to the sampler jet and recirculate the sampler for 5 minutes.

4. Turn off the air and open the door; survey the opening.

5. Remove the sample bottle from the sample jet station with tongs. Pour the contents of the sample bottle into the composite bottle. Discard the sample bottle and close the door.

6. When a composite sample is to be submitted, check with supervision for the sample code and prepare a sample bottle.

7. Using a bulb pipette, transfer 10 milliliters of solution from the composite bottle to the sample bottle.

8. Insert the bottle into the sample carrier and close the carrier. Wrap the carrier in plastic and check with supervision for tags and request sheets.

9. Transfer the sample to the 3019 Analytical Lab (see Section E below).

E. Transfer of Samples to Building 3019 Analytical Laboratory

1. Check that the sample is in the carrier, wrapped in plastic, and that the radiation tags and transfer tags are filled out and attached.

2. Notify supervision that the sample is ready to be transferred. Supervision will inform the Analytical Laboratory.

3. Set the sample carrier in the sample transfer box on the large truck. Close the lid and secure the latches.

4. Transfer the carrier by truck to the Building 3019 loading dock (west end). Personnel at Building 3019 will receive the carrier and remove the sample bottle.

5. When the sample has been removed, check that the carrier is properly tagged and return it to FPDL.

F. Special Sampling Procedure for Crystallizers (Cell 18)

NOTE: The method for withdrawing samples from the crystallizers (C17, C27, C38, C48, and P45) is different from the sampler method described in Section A above. Samples may be withdrawn for crystallizers either for delivery to Analytical or for process checks. The following points apply to all crystallizer sampling.

1. Always double-check that the agitator in the vessel to be sampled is on.

2. Never sample a crystallizer (for crystal bed size) while it is cold if the crystal bed size as determined by temperature break is greater than 10% of the solution volume.

3. Do not let the crystallizer sample flask recirculate unless so instructed by supervision.

4. Do not turn on steam to sampler jackets unless so instructed by supervision.

5. Prepare for crystallizer sampling as follows:

   a. Check that the sampler flask is in place, that the sampler flask off-gas nipple is connected to the off-gas (middle) line of the sample station, that the water addition (east) line is connected to the sample flask, and that both stopcocks on the flask are closed.

   b. Close the off-gas (middle) line on the spare sample station with a blank ball-joint.

   c. Check that a blank male ball-joint is ready for closing the sampler flask vent nipple.

   d. Connect the bottom inlet of the sample flask to the sample nipple from the vessel to be sampled.

   e. Check that the manual valve from crystallizer jackets to condensate drain (on east side of Cell 15) is closed, and check that all process water and steam valves to sampler jackets are closed.

6. Sample crystallizers while they are cold (for crystal bed size determination) as follows:

   a. Check that the agitator to the vessel to be sampled is on.

   b. Open the process water to the sampler jacket of the vessel to be sampled.

      NOTE: P713 does not have a jacket.

c. Open the bottom stopcock to the sampler flask.

d. Hold the blank male ball-joint with the manipulator so that it can be inserted and removed from the vent nipple easily.

e. Turn on the sampler jet and set the blank ball-joint on the vent nipple (do not clamp).

f. Pull ~500 milliliters of slurry into the sampler flask; then immediately turn off the sampler jet and remove the blank ball-joint from the vent.

g. Let the slurry flow back to the vessel. Wait 30 seconds after the flask is empty and then repeat Steps d through g twice.

h. Turn on the sampler jet, close the vent and pull ~500 milliliters of slurry into the flask, and then immediately close the bottom stopcock.

i. Turn off the sampler jet and vent the flask.

j. Let the crystals settle for 5 minutes.

k. Measure the total volume in the flask and measure the crystal volume in the flask. Calculate the percent of crystals.

l. Turn on the sampler jet and close the flask vent.

m. Open the bottom stopcock.

n. As soon as the crystals are thoroughly mixed, turn off the sampler jet and vent the flask.

o. Let the slurry flow back to the vessel. Rinse the flask immediately with ~2 liters of water. (Add the water through the water inlet line to the flask while the bottom stopcock is still open.)

p. Close the bottom stopcock.

q. Turn off the cooling water to the sampler jacket.

7. Sample crystallizers hot (for gamma chamber checks) as follows:

a. Prepare a sample bottle calibrated at 10-milliliter level (also a marked sample bottle for Analytical if required).

b. Check that the agitator of the vessel to be sampled is <u>on</u>.

c. Open the <u>hot</u> process water to the sampler jacket.

d. Open the bottom stopcock to the sampler flask.

e. Hold the blank male ball-joint with the manipulator so that it can be inserted and removed from the vent nipple easily.

f. Turn on the sampler jet and set the blank ball-joint on the vent nipple (do not clamp).

g. Pull ~500 milliliters of solution into the sampler flask; then immediately turn off the sampler jet and remove the blank ball-joint from the vent.

h. Let the solution flow back to the vessel. Wait 30 seconds after the flask is empty and then repeat Steps f through h twice.

i. Turn on the sampler jet, close the vent and pull ~500 milliliters of solution into the flask, and then immediately close the bottom stopcock.

j. Turn off the sampler jet and vent the flask.

k. Holding the sample bottle under the flask side arm, open the stopcock and collect 10 milliliters of solution and then close the stopcock.

l. Open the bottom stopcock of the sample flask and allow the solution to drain back to the vessel. Rinse the flask immediately with ~2 liters of water (add the water through the water inlet line while the bottom stopcock is open).

NOTE: If there are solids in the solution, the sample flask will be agitated as in Steps 6.l through 6.n above before letting it drain back to the vessel.

m. Close the bottom stopcock.

n. Turn off the hot process water to the sampler jacket.

G. Sampling of Special Vessels

1. Vessels R111 and R18 are vacuum tanks and the sample inlet and return lines to these tanks are valved. Always check that the tank is open to off-gas and that both valves on the sampler lines are open before attempting to sample these vessels.

2. Tank E16 has a manual valve on the sample intake. This valve is operated from the Cell 18 window. Always open this valve before attempting to sample E16 and close it after sampling is complete.

H. Gamma Chamber Measurements of ^{137}Cs Samples

1. Before making a gamma chamber measurement, check that the sample dolly is in the blister at the full-stop position. Measure the background on the gamma chamber and note it for use at Step 5 below.

2. Pull the sample dolly handle slowly into Cell 18 until the cup is exposed.

3. Set the sample bottle (be sure that the cap is secure) into the cup.

   NOTE: Sample volumes will be 10 milliliters unless otherwise specified.

4. Slide the dolly slowly into the gamma chamber.

5. Read the gamma chamber dial and refer to the calibration chart to determine the ^{137}Cs concentration in the sample.

   NOTES:

   a. The gamma chamber is calibrated for 10-milliliter samples of ^{137}Cs. The calibration charts give the dial reading versus millicuries/milliliter (mc/ml) of ^{137}Cs. Be sure that the chart you use corresponds to the scale setting on the instrument.

   b. If the sample volume measured is less than 10 ml, a correction will have to be made. The correction factor is 10 divided by the actual sample volume. For example, if a 5-ml sample shows 2 mc/ml, the actual concentration is

   $$2 \times \frac{10}{5} = 4 \text{ mc/ml}.$$

   c. Always determine the mc/ml first; then convert the background reading to mc/ml and subtract it from the measured value.

6. After the measurement has been made, pull the sample dolly slowly into Cell 18, remove the sample from the cup, and slide the dolly back into the blister.

FISSION PRODUCTS DEVELOPMENT LABORATORY

SOLID WASTE DISPOSAL

I. Nonradioactive Solid Waste

A. Burnable Waste - Brown Dumpster

1. Put nonradioactive burnable waste (such as waste paper and boxes) in the brown Dempster Dumpster located at the northwest corner outside Building 3517. This Dumpster is picked up and dumped routinely.

2. Do not put nonburnable waste in the brown Dumpster. See below for disposal of nonburnables.

B. Nonburnable Waste - Yellow Dumpster

1. Put small items such as glass reagent bottles (after being rinsed) or small metal pieces in the yellow Dumpster. Larger volumes of nonburnable waste are handled by special pickups.

C. Waste Chemicals and Solvents

1. Do not place chemicals or solvents in the Dumpsters. They will be handled by special pickup.

   Any waste material being removed from a contamination zone is assumed to be contaminated and is disposed of under the radioactive waste procedures below.

II. Low-Level Solid Waste (Yellow Dumpster)

A. Definition

Low-level waste consists of disposable items with radiation readings low enough to permit their disposal in the yellow Dumpster. These materials include paper, plastic, wipes, etc., used in contamination (C) zone work, clothing too highly contaminated to be sent to the laundry, tools that cannot be repaired, and similar items. The upper limit for an individual item to be classed as low-level waste is 200 mr/hr; the upper limit for a container of waste to be considered as low level is 6 mr/hr.

B. Procedure

1. Place low-level wastes in yellow hot cans, specially marked waste cans, or marked plastic bags. These containers are located at the zone portals of C zones and at other selected spots. At these areas are also located containers for gloves, shoe covers, and coveralls.

2. When a disposable item is no longer in use, survey it and place it in a waste container if its maximum radiation reading is less than 200 mr/hr. If an item reads greater than 200 mr/hr, contact supervision for special handling instructions.

3. Survey low-level waste containers periodically during operations. If a container exceeds 6 mr/hr, it will be transferred to the yellow Dumpster as in Step 4.

4. When a container is to be transferred to the yellow Dumpster, tape the plastic bag closed. Remove the bag from the can and transfer it to the yellow Dumpster.

5. If the low-level waste items are bulky (such as blotter paper from C zones), roll them, place in plastic bags, seal, and transfer to the Dumpster.

6. Always check with supervision before disposing of hardware such as tools and hoses.

7. Do not leave the covers of the yellow Dumpster open except when putting materials into it.

8. When the Dumpster is full, tape the lip with masking tape and contact Health Physics for survey and tagging.

C. General Information

1. Clothing (gloves, coveralls, shoe covers, plastic suits) is considered disposable if it reads greater than 20 mr/hr. If less than 20 mr/hr, these items are placed in specially marked containers for transfer to the laundry.

2. All low-level waste containers are emptied on a routine basis on the 4 to 12 shift.

3. Supervision will contact the burial ground for pickup.

III. High-Level Solid Waste

A. Bottom-Loading Can Shield

NOTE: This shield is used to load contaminated solid waste from Cells 18 and 19. The shielded can will be removed to the burial ground by the riggers.

1. Packaging

a. Put a plastic bag liner inside a 1-gallon waste can. Tape the liner at several points to prevent it from crumpling.

b. Insert the can into the cell by opening the sample blister and placing the can on the dolly. Monitor the blister opening during the operation; wear gloves and assault mask.

c. Close the blister top and pull the can into the cell.

d. Clean waste to be discarded as well as possible inside the cell. Wash glassware thoroughly; dissolve solids, precipitates, etc., and send to chemical hot waste if possible; otherwise, leach them as instructed by supervision. Empty reagent bottles and rinse. Do not add bottles containing liquids to the waste can.

e. Load the waste material into the can, being careful not to damage the plastic liner.

NOTE: Material should be compacted into the smallest possible volume. Small pieces of material (sample bottles, caps, centrifuge tubes, etc.) can be placed inside beakers or other larger vessels. Paper and fabric can be folded and pressed down.

f. When the can is full, fold the plastic over the material and set the lid on the can. Press the lid down firmly.

g. Set the can on the dolly.

2. Loading of Shield

a. Check that the shield is empty and ready to be used.

b. Remove the large top plug from the sample blister.

c. Using the hoist, set the shield over the blister opening. Release the latch and pin. Withdraw the bottom drawer to the end of the track.

d. Pull the can from the cell into the blister by means of the dolly.

e. Insert the lifting rod through the top of the shield and engage the hook into the bale on the can.

f. Lift the can into the carrier. Survey the shield. If the reading is greater than 200 mr/hr, lower the can into the blister and notify supervision.

g. Slide the bottom drawer under the can and secure the latch and pin.

h. Release the hook from the can and remove the lifting rod. Survey the lifting rod and decontaminate it if necessary.

i. Obtain Health Physics survey of shield. Clean to <1-mr/hr smear level.

j. Cover the shield with plastic and attach material transfer tag.

3. Transfer

Supervision will contact the burial ground and arrange for the transfer. Burial ground supervision will handle the transfer, dumping, and return of the shield.

B. One-Inch Lead-Lined Dempster Dumpster

This carrier is used to load relatively low-level contaminated solid waste from the manipulator cells. Supervision will determine when the Dumpster is to be used.

1. Packaging

a. Load waste to be put in the Dumpster into cans as in Section II.B.2 above. Place bulk solids (wipes, paper, manipulator boots, plastic) in plastic bags. The cans and bags will be accumulated in Cell 14 for loading into the Dumpster.

2. Loading of Dumpster

a. Check that the Dumpster is empty and ready to be used.

b. Open Cell 15 top and lower the Dumpster into the cell.

NOTE: This Dumpster is a close fit in Cell 15, and careful operation is necessary while it is being inserted. The Dumpster must be positioned so that the top will open toward the west or north.

c. Attach chokers to the Dumpster lid through the center block and with the crane raise the lid to provide opening for loading. Leave the lid suspended from the crane. Position plastic shields over the Cell 15 top. Rope off the area around Cell 15 top as a radiation zone.

d. Mount a radiation detection device behind the Dumpster so that it can be seen from Cell 15 window.

e. Transfer waste material from Cell 14 to Cell 15, one item (bag or can) at a time.

f. Lower the waste into the Dumpster, using the manipulators; do not release until the radiation reading is taken. If an item (can or bag) causes the radiation reading to go above 200 mr/hr through the Dumpster, withdraw the material, return it to Cell 14, and contact supervision.

g. When all waste has been loaded into the Dumpster, close the lid.

h. Enter Cell 15 under Cell Entry regulations and survey the Dumpster. If it reads greater than 200 mr/hr, contact supervision. Decontaminate the Dumpster.

i. Remove the Dumpster from Cell 15 and decontaminate to less than 1-mr/hr smear level. Wrap the Dumpster in plastic. Attach material transfer tag.

3. Transfer

Supervision will contact the burial ground and arrange for the transfer. Burial ground supervision will handle the transfer, dumping, and return of the Dumpster.

C. Off-Gas Filter Shield

See "Cell 20 (Hot Off-Gas) Operation."

D. Cell Ventilation Filter Shield

See Cell Ventilation Filter Change Procedures.

E. Three-Inch Stainless Steel Dumpster

This carrier is used to remove moderate-level contaminated waste from the manipulator cells. Supervision will determine when this Dumpster is to be used and will also establish the maximum permissible terminal radiation reading through the carrier after it is loaded.

1. Check that the heat sealer is in place on the east side of Cell 15. Insert a sufficient number of plastic bags (check with supervision) to complete the loading. These bags have tabs for easy opening; leave all bags in Cell 15 until ready to begin loading.

2. Before placing the Dumpster in Cell 15 prepare it as follows:

NOTE: The Dumpster is assumed to be empty, not cleaned, wrapped in clean plastic, and in the carrier rack at the first level west of Building 3517.

a. Obtain from FPDL supervision the key for unlocking bar across the top of Dumpster. Then lift the Dumpster with the short, heavy, 3-hook (using only two) choker and place it on clean paper in a roped-off area in the crane bay area near Cell 15.

b. With a Health Physics representative present, carefully remove the plastic and dispose immediately. Health Physics will then determine the level of contamination of the Dumpster. If FPDL supervision and the Health Physics representative decide that the contamination is too high for the Dumpster to be cleaned in

the open crane bay area, place it in Cell 15 or Cell 1 (or suspend it just below the top of Cell 15 or Cell 1) and clean to a level of contamination which will permit further cleaning in the crane bay area. Then return the Dumpster to the paper in the roped-off area of the crane bay and further clean to a smear tolerance of <1 mr/hr.

c. Using special plastic bags (check with supervision), wrap the Dumpster securely so that the plastic will hold fast while the lid is being raised and the Dumpster is being loaded. Tape the eyelets for the chokers or hooks.

3. Place the Dumpster in Cell 15 as follows:

a. Remove the Cell 15 large block and set the Dumpster on the large dolly track in Cell 15 so that the lid raises away from the transfer tray. Then replace the Cell 15 block and attach a long choker to the lid through the hole provided by removing the small plug in the Cell 15 block.

NOTE: Do <u>not</u> place the Dumpster in Cell 15 until <u>all</u> material to be loaded has been prepared for loading and accumulated in Cell 14.

b. Mount a radiation detection device (a cutie pie double-bagged in plastic) on the side of the carrier that is visible from the Cell 15 window.

NOTE: It may be desired to attach this instrument prior to placing the Dumpster into the cell. It is also possible to insert the cutie pie in the cell, and obtain readings by holding the cutie pie with the manipulator hands.

c. Insert into the cell a plastic liner for the Dumpster.

4. Load the carrier as follows:

a. Raise the lid of the Dumpster by use of the long choker and the 20-ton crane; leave the lid suspended by the crane.

NOTE: The lid may be secured by putting a bar through the loop on the choker if supervision so directs.

b. Insert the plastic liner into the Dumpster and tape it so that it will remain in place while the Dumpster is being loaded but can be loosened and closed by use of a drawstring when the loading is completed.

c. Check with supervision for approval to begin loading.

d. Install one plastic bag in the bag holder. The jaws of the heat sealer should be open.

e. Transfer one plastic bag into Cell 14W.

f. Insert a waste can into the plastic bag in Cell 14W.

g. Transfer the bagged waste can into Cell 15.

h. Insert the bagged waste can into the plastic bag that is in the bag holder.

i. Pull the top of the bag together and close the jaws of the heat sealer around it.

j. Turn on the power to the heat sealer. Power pack settings are _____and_____(check with supervision).

k. After power is off, allow the plastic to cool for 10 seconds before opening the jaws of the heat sealer.

l. Using the extended-reach manipulator, open the jaws of the heat sealer and remove the bagged waste from the bag holder.

m. Transfer the bagged waste directly into the Dumpster. If the radiation reading on the Dumpster exceeds 500 mr/hr, contact supervision.

n. Repeat Steps d through m for each waste can until the Dumpster is full or all cans have been loaded.

o. Close the plastic liner by pulling the drawstring tight and knotting it twice.

p. The Dumpster is now ready to be smeared. Make up 12 smears by taping blotter paper on about one-half of a 16-inch wood splint. Punch a small hole in the uncovered end and tie a string through it. Smear as follows:

   (1) Insert a smear through the small center plug of Cell 15 top. Lower the smear into the cell by the string.

   (2) Using the extended-reach manipulator, grasp the smear by the uncovered end.

   (3) Smear the first area listed below. Pull the smear to the cell top and read it with a survey meter. Discard the smear and repeat the operation for the other areas, in the order listed.

Smear Number	Area*
1	Left lip
2	Center lip
3	Right lip
4	Left inside surface
5	Center inside surface
6	Right inside surface
7	Left inner top
8	Right inner top
9	Left side of plastic bag
10	Left side of plastic bag closure
11	Right side of plastic bag closure
12	Right side of plastic bag

*As the area appears when looking into the front of the Dumpster.

(4) Using damp swabs, clean areas 1-8 to less than 1-r/hr smear levels.

(5) If an individual smear reads greater than 1 r/hr, stop the smearing operation and contact supervision.

(6) Report final smear results to supervision.

(7) Lower the Dumpster lid.

5. Prepare the carrier for transfer to the burial ground as follows:

a. Remove choker from the lid, replace plug in the Cell 15 block, and remove the large Cell 15 block.

b. With FPDL supervision and Health Physics representative present, raise the carrier to just below the Cell 15 top and ascertain whether the radiation level will permit the carrier to be removed for transfer.

c. If the radiation level is within tolerance, remove the plastic from the Dumpster and carefully bag the contaminated plastic or drop it into Cell 15. Remove tape from the Dumpster and clean it while it is over Cell 15 (if necessary) until Health Physics determines that the carrier may be removed to an adjacent roped-off area for further cleaning. The Dumpster should then be cleaned as quickly as possible (due to radiation through the Dumpster) to ~1-mr/hr smear tolerance.

d. Then wrap the Dumpster in clean plastic and place it in the carrier rack at the first level west. Insert the locking bar and lock it.

The Health Physics representative will then survey the carrier, attach the material transfer tag, and complete his portion of the Burial Ground Transfer Request. FPDL supervision will complete the Burial Ground Transfer Request and attach it to the outside of the Dumpster.

FPDL supervision will contact the burial ground and arrange for the transfer. Burial ground supervision will handle the transfer, dumping, and return of the carrier.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CELL 20 (HOT OFF-GAS) OPERATION

NOTE: The off-gas scrubbers, N120 and N220, will be operated continuously except when supervision instructs otherwise. Scrubber solutions will be changed once per day on the 8 to 4 shift.

B120 and B220 are parallel filter banks in the building off-gas header. Each bank contains a Neva-clog (stainless steel) roughing filter and a CWS (paper) absolute filter. One bank of filters must be in operation at all times to maintain off-gas. Neva-clog filters will be washed and CWS filters changed as instructed by supervision.

I. Changing Scrubber Solutions

A. Changing N120 Scrubber Solution

1. Determine that the liquid level of M5 is 10% or greater. If below 10%, pump 60% HNO_3 to M5. Do not exceed 80% liquid level in M5.

2. Turn off the N120 recirculation pump. Leave the panelboard valve to the pump open so that the heat exchanger unit will drain.

3. Sample N120 if requested. Code_____Sc 120.

4. Open the block valve and turn on the jet from N120 to S324. While jetting, turn on water spray to N120. Set rotometer at 50% and spray for 3 minutes; then turn off the spray. When N120 is empty, turn off the jet and close the block valve.

5. Close the panelboard valve to the recirculation pump. Open the prime water valve and backwash to 10% liquid level in N120; then close the prime water valve.

6. Open the block valve and turn on the jet from N120 to S324. When N120 is empty, turn off the jet and close the block valve.

7. Turn on water spray to N120. Set rotometer at 50%. Add water to 20% liquid level in N120; then turn off the spray.

8. Open the valves from M5 to N120 and add 7 gallons of 60% HNO_3. Close valves from M5 to N120.

9. Turn on water spray to N120, set rotometer at 50%. Add water to N120 until the liquid level reaches 50%. Turn off the water spray.

10. Open the panelboard valve to the N120 recirculation pump. Turn on the N120 recirculation pump and check flow rate.

NOTE: It may be necessary to prime the pump by means of the prime water line at the Cell 20 north wall. Do not run Chem Pumps without flow.

11. After the N120 liquid level is steady, turn on water spray N120. Set rotometer at 50%. Add water to N120 until the liquid level reaches 50%; then turn off the water spray.

B. Changing N220 Scrubber Solution

1. Turn off the N220 recirculation pump. Leave the panelboard valve to the pump open so that the heat exchanger unit will drain.

2. Sample N220 if requested. Code_____Sc 220.

3. Open the block valve and turn on the jet from N220 to S324. While jetting, turn on water spray to N220. Set rotometer at 50% and spray for 3 minutes; then turn off the spray. When N220 is empty, turn off the jet and close the block valve.

4. Close the panelboard valve to recirculation pump. Open the prime water valve and backwash to 10% liquid level in N220; then close the prime water valve.

5. Open the block valve and turn on the jet from N220 to S324. When N220 is empty, turn off jet and close block valve.

6. Turn on the water spray to N220. Set rotometer at 50%. Add water to N220 until the liquid level reaches 50%. Turn off the water spray.

7. Open the panelboard valve to the N220 recirculation pump. Turn on the N220 recirculating pump and check the flow rate.

NOTE: It may be necessary to prime the pump by means of the prime water line at the Cell 20 north wall. Do not run Chem Pumps without flow.

8. After the N220 liquid level is steady, turn on water spray N220. Set the rotometer at 50%. Add water to N220 until liquid level reaches 50%. Turn off the water spray.

II. Washing of Stainless Steel Filters (B120S or B220S)

A. Water Wash

NOTE: This procedure assumes that filter bank B120 is on stream and B220 is on standby. If B220 is on stream and to be washed, reverse the filter designations in the procedure.

1. When B120S is to be washed (supervision will determine), remove the small lead plugs at the top of Cell 20 and operate the manual valves as follows, using the special wrench:

   a. Open the valve from the scrubbers to B220.

   b. Open the valve from B220 to the stack.

c. Close the valve from B120 to the stack.

d. Close the valve from the scrubber to B120.

2. At first level north, connect water line by Chicago coupling to line B120W. Open manual valves and allow water to run for 2 minutes.

3. Turn off manual valves. Disconnect Chicago coupling.

4. Allow filter to drain for at least 15 minutes.

5. At the top of Cell 20, operate the valves as follows:

a. Open the valve from the scrubbers to B120.

b. Open the valve from B120 to the stack.

c. Close the valve from B220 to the stack.

d. Close the valve from the scrubbers to B220.

e. Insert the lead plugs.

B. Chemical Wash

1. Switch off-gas to B220 bank as described in Section II.A.1 above.

2. Set up in Area 34 as follows:

a. Close all valves in the acid lines.

b. Set up a 55-gallon stainless steel drum. Place Tygon hose to the acid pump in the drum.

c. Set valves to pump from M1134 to the drum.

d. Add 45 gallons of water to the drum.

e. Pump 5 gallons of 60% HNO_3 into the drum. Close valves.

f. Adjust valves to pump from the drum to second level east.

3. Set up at second level east as follows:

a. Connect Chicago coupling on line from acid pump (Area 34) to second level east with Chicago coupling on B120S chemical wash line by Tygon tubing.

b. Close valve to M120 fill leg and open valve to Chicago coupling on line from acid pump to second level east.

c. Open valve to B120S chemical wash line.

4. Pump 25 gallons of solution from the drum in Area 34. Turn off pump.

5. Close valve to Chicago coupling on line from acid pump to second level east and close valve to B120S chemical wash line.

6. Water wash B120S as described in Section II.A above.

7. Allow B120 bank to dry for 15 minutes; then switch off-gas from B220 bank to B120 bank as described in Section II.A.5.

8. Switch Tygon from B120S chemical wash line to B220S chemical wash line.

9. Open valve to Chicago coupling on line from acid pump to second level east and open valve to B220S chemical wash line.

10. Pump remainder of solution from the drum in Area 34. Turn off pump.

11. Close valve to Chicago coupling on line from acid pump to second level east and close valve to B220S chemical wash line.

12. Water wash B220S as described in Section II.A.

13. Open valve to M120 fill leg.

14. Set valves in Area 34 to pump acid from M1234 to Areas 31-32. Rinse the 55-gallon drum and store in the solvent storage shed.

III. CWS Filter Change

NOTE: This procedure assumes that the CWS filter in B120 bank is to be changed and that B220 bank will be on-line during the filter change. If B220P is to be changed, reverse the filter designations.

A. Preparation

1. Set up second level east as a Radiation/Contamination Zone. Cover Cell 20 (except B120 filter shield), aisleway, rails, and east side of bay wall with paper.

2. Paper down outside north door to aisleway. Set up supply of safety equipment and provide bags for disposing of used safety equipment.

3. Set up the following equipment at Cell 20:

   a. New CWS filter. Check that filter has strap with rod nuts welded to it and that strap and nuts are properly attached. Check that the gasket is firmly cemented on the sealing face and that there are no cracks in the gasket. The filter element should be in perfect condition.

b. B120 filter can with gasket cemented firmly to base. Check that there are no cracks in the gasket.

NOTE: B120 and B220 filter cans are not interchangeable.

c. Filter can cover with four lock bolts.

d. Filter shield base.

e. Tools:

8-inch crescent wrench	1 spreader hook
10-inch pipe wrench	1 wrench for filter can cover lock bolts
hammer	duct-seal
2-eye wrenches	plastic bags
4 rod pins	

f. Auxiliary armor plate.

g. Radiation detection instruments.

NOTES: Supervision will fill out a Radiation Work Permit and arrange for Health Physics coverage.

Supervision will issue instructions for turning on chilled water to all tanks containing radioactive solutions and stopping all processing that involves off-gas.

4. Switch to B220 filter bank and wash B120 Neva-clogs.

B. Removal of Used CWS Filter

1. Loosen the holding clamps and remove the eight plugs in the top of the filter shield.

2. Move the lock rod on the filter shield to the "out" position.

3. Attach eye wrenches to the 2 wedge rods (second pair of rods from west side of carrier). Pull up to the first holes in the rods (manually if possible; otherwise connect the eye wrenches to the crane, using spreader hook to prevent bending the rods). Insert pins in the first holes.

4. Attach the 2-eye wrenches to the filter pull rods (the second pair of rods from the east side of the filter shield). Connect the eye wrenches to the crane, using spreader hook, and lift the filter until it is free of the wedge. Shake the wedge if necessary to free it from the filter.

5. Insert pins into the second holes of the filter rods.

6. Remove the filter can hold-down rods at the corners of the filter shield by turning counterclockwise.

7. Remove the pins holding up the wedge and lower the wedge. Remove the rods. (Careful - Do not damage the stainless steel rod threads.)

8. Raise the filter rods to the level of the third holes in the rods and pin in position. (It will probably be necessary to use the spreader hook and crane.)

9. Lock the filter can into position by pushing the lock rod on the filter shield to the full "in" position.

10. Disconnect the crane from the filter rods.

11. Position the filter can cover on the filter shield base to fit the bottom of the filter can. Lift the filter shield from the 4 corner eyes. (Use the 4 short chokers with shackles.)

12. Position the filter shield over the filter shield base. Lower the shield so that the filter can sets in the can cover. (The edge of the can will hang below the edge of the filter shield, thus giving enough space to lock the cover to the can.)

13. Lock the cover onto the filter can.

14. Set the filter shield onto its base and lock it with the 4 lugs.

15. Place the auxiliary armor plate in position on the side of the filter shield.

16. Remove the filter rods. Replace the plugs in the top of the filter shield and tighten the holding clamps.

17. Decontaminate the filter shield for transfer to burial ground. Wrap in plastic and request Health Physics survey. Supervision will fill out appropriate transfer papers.

C. Installation of New CWS Filter

1. Remove all loose debris from the plenum. Transfer the contained debris to the yellow Dumpster. Decontaminate the filter plenum by hosing down with water. Allow plenum to dry before installing the filter.

2. Reinspect the new CWS filter. Check to see that the gasket is firmly cemented on the sealing face and that there are no cracks in the gasket. Check that the filter element is in perfect condition.

3. Place the CWS filter in position by hand. Make certain that the gasket is properly fitted and unbroken.

4. Place the wedge in position by hand. See that it is pushed all the way down and that it holds the filter firmly in place.

5. Align the filter can properly on its base gasket (use the crane).

6. Install the 4 filter can hold down rods. Tighten the rods evenly.

7. Install the 2 filter lifting rods. Install the 2 wedge lifting rods.

8. Make the 4 holes at the top of the filter can leaktight by sealing around the lift rods with Duct-Seal.

9. Switch off-gas to the B120 bank and check for leaks:

10. Set the filter shield over the filter can (use the crane).

11. Place the plugs in the top of the filter shield and tighten the holding clamps in position over the plugs.

12. Decontaminate the area. Health Physics will check personnel and clear the Radiation/Contamination Zone.

D. Dioctylphosphate (DOP) Test of Cell 20 CWS Filters

NOTE: DOP tests are made by P&E personnel. Chemical operator support will be needed in hooking up the smoke generator at first level east and in decontamination.

Supervision will make out a Radiation Work Permit for opening the door to Cell 20 scrubber room and arrange for Health Physics coverage

1. Operator will set up a Contamination Zone at the door to Cell 20 scrubber room.

2. With Health Physics personnel present, operator will carefully open the door to Cell 20 scrubber room.

3. Operator will connect the coupling from the smoke generator to the inlet line, without entering the cell.

4. After P&E personnel have taken their readings, operator will disconnect the smoke generator.

NOTE: Health Physics will check personnel and clear the zone.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CELL VENTILATION CONTROL

A. Cell Ventilation Adjustment

1. Cell Ventilation Filter House: There are three dampers on the main cell ventilation house: one on the east bank, one on the center bank, and one on the west bank. Two of the three banks will be on stream at any time (supervision will designate which ones will be on stream). The dampers will be adjusted during normal operation to maintain between 1.5 and 2.0 inches of negative pressure in the manipulator cells. Manipulation of these dampers should be done slowly and with only one of the dampers at a time in order to prevent pressure changes in the cell ventilation system.

   The dampers in the cell ventilation house will be closed during the washing of the cell ventilation filter pit roughing filters and during the changeout of filters in either the cell ventilation filter house or pit.

2. Bypass Filter Pit: The damper on the outlet from the bypass filter is used to provide some cell ventilation during washing or filter replacement in either the main filter pit or house.

3. Cell Ventilation Filter Pit: There are no control dampers in the cell ventilation filter pit. This filter system is used in series with the CWS absolute filter system in the cell ventilation house to provide additional filtration of the cell ventilation air going to the stack.

4. Manipulator Cell Ducts: There are no damper controls in the outlet ducts from the manipulator cells. These ducts may from time to time be partially closed by placing covers over the in-cell CWS filter inlets. This would be done only to control an individual cell air flow and pressure at specified conditions.

5. Process Cell Ducts: The damper controls on the outlet ducts from Cells 2 through 9 are located in the main service tunnel. The dampers can be used to make final adjustments on the cell ventilation to the process cells.

6. Measurements: The cell ventilation pressures on the manipulator cells (Cells 10 through 15) are indicated on the cell ventilation panelboard located in the manipulator area. A Magnehelic gage is located on the east face of Cell 16 to measure the process cell ventilation pressure. Cell 1 pressure is measured on the Cell 1 panelboard. The cell ventilation pressures on Cells 16, 18, and 19 are measured with gages on the face of each of the cells.

Under normal operating conditions the cell ventilation pressures on all cells are kept at negative pressures of 1.5 to 2.0 inches of water. The alarm points are set at -0.5 inch. The pressure drop across the cell ventilation pit and house CWS absolute filters and roughing Neva-clog filters is indicated by gages located on the cell ventilation house. The main cell ventilation duct pressure and the pressure between the cell ventilation house and duct are also measured with gages located on the cell ventilation house.

B. Washing of Neva-Clog Filters in the Main Filter Pit

Check with supervision before washing. Supervision will schedule operations and will determine which type of washing, water or chemical, will be done. If water washing is to be done, proceed as outlined in Steps 1 through 11 below. If chemical washing is to be done, proceed as outlined in Steps 12 through 29 below.

1. Connect a water hose to the hot water outlet on the east side of the building and to the south spray connection at the filter pit. Open both the north and south manual valves.

2. Open the bypass cell ventilation filter damper.

3. Close the outlet with dampers from the main cell ventilation house. Check with supervision to determine what steps should be taken to maintain a minimum of 0.25 inch negative pressure in the manipulator cells.

4. Turn on the hot water and spray the south side of the Neva-clog filters for 10 minutes.

5. Turn off the water and connect the water hose to the north side of the Neva-clog filters.

6. Turn on the water and spray the north side for 10 minutes.

7. Disconnect the hose, close both manual valves to the Neva-clog filters, and allow the system to drain for 10 minutes.

8. NOTE: Check that the cell ventilation sump pump is operating during Steps 4 through 6 above.

9. Open the cell ventilation house dampers (supervision will designate which two banks of filters are to be placed on stream) slowly and adjust them so that the pressure in the manipulator cells is at least a negative 2.0 inches of water.

10. Close the bypass cell ventilation damper.

11. Readjust the filter house dampers as necessary to obtain a negative pressure of between 1.5 and 2.0 inches of water in the manipulator cells.

12. Set up a stainless steel 55-gallon drum near the M-1134 HNO_3 acid storage tank.

13. Add 45 gallons of water to the drum.

14. Pump 5 gallons of 60% HNO_3 from M-1134 to the 55-gallon drum.

15. Connect a hose from the pump discharge line to the south side of the cell ventilation pit Neva-clog filter. Open the manual valve on the south side spray system.

16. Open the bypass cell ventilation filter damper.

17. Close the outlet dampers from the main cell ventilation house. Check with supervision to determine what action should be taken to maintain a minimum negative pressure of 0.25 inch of water in the manipulator cell.

18. Close all valves at the pumping station that go to the building proper; open the pump discharge valve.

19. Place a dipstick in the 55-gallon drum and connect it to the pump suction line.

20. Pump approximately 25 gallons from the stainless steel drum through the south side spray system. Turn off the pump.

21. Carefully, using chemical handling precautions, disconnect the hose from the south side of the Neva-clog and connect it to the north side of the Neva-clog filter spray. Close the manual valve on the south side spray system.

22. Open the north side spray system manual valve.

23. Turn on the pump and pump the remaining solution through the north side spray system.

24. Carefully disconnect the hose from the pump discharge and close the manual valve on the pump discharge line that goes to the cell ventilation spray system.

25. Give both sides of the Neva-clog filter a water wash as described in Steps B.4 through B.8 above.

26. Open the cell ventilation house dampers (supervision will designate which two banks of filters are to be placed on stream) slowly and adjust them so that there is a negative pressure in the manipulator cells of at least 2.0 inches of water.

27. Close the bypass cell ventilation damper.

28. Readjust the filter house dampers as necessary to obtain a negative pressure of between 1.5 and 2.0 inches of water in the manipulator cells.

29. Rinse out the 55-gallon stainless steel drum and the hose with water. Store these items.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CELL VENTILATION FILTER CHANGE

FILTER HOUSE

The replacement of the CWS filters in the main filter house requires a complete shutdown of the FPDL. Supervision will schedule the changeout and issue specific instructions for the performance of the steps of the following procedure. A filter changeout requires full-time FPDL supervision.

I. Preoperational Check

A. FPDL

FPDL personnel will be responsible for the following:

1. Closing all cells and sealing with tape.
2. Containing the master ends of all the manipulators with plastic bags.
3. Turning off all steam headers.
4. Turning off all in-cell lights.
5. Putting down paper and roping off the area directly in front of the filter house doors.
6. Setting up an entry and exit portal.
7. Providing the following protective clothing and supplies:

Coveralls	Blotting paper
Gas masks	Rope
Shoe covers	Sheet plastic
Gloves	Plastic bags
Masking tape	Contamination cans

8. Having hot truck available.
9. Having Inspection Engineering personnel on standby to DOP (di-octylphosphate) test the filters while they are being installed.

B. Health Physics

Health Physics personnel will be responsible for:

1. Checking the CAM's in the building.

2. Supplying dosimeters and film rings.

3. Supplying surveying equipment.

C. Supervision

Supervision will arrange for fabrication of the following special equipment before time for the filter change:

1. Special wooden boxes for holding the filters as they are being removed.

2. Wooden sheets for sealing the filter from the bypass cell ventilation system.

3. Wooden table for placing the wooden box on during withdrawal of the filters.

4. Plastic bags for the wooden boxes.

II. Operation (FPDL personnel)

A. Bypass Filter Station

1. Open the cell ventilation bypass filter damper.

2. Slowly close the cell ventilation house dampers.

NOTE: Supervisor must direct the necessary in-building action, such as placing plastic over the cell inlet filters, bagging of the master ends of the manipulators, etc., to maintain a minimum negative pressure of 0.25 inch of water in the cells while the building is on the bypass system.

B. Filter Removal

1. East Bank CWS Filters

a. Lay blotter paper and plastic in front of the cell ventilation house doors for a distance of 10 to 12 feet south of the doors.

b. Open the east top door.

c. Open the east bottom door.

d. Slide the wooden sheet in the opening through the bottom door.

e. Place the table against the south face of the house directly in front of the east door.

f. Place the plastic bag on the wooden box.

g. Position the wooden box on the table directly in front of the east filter door opening.

h. Tape the plastic bag to the cell ventilation housing.

i. Open the screw-down rods that are located on top of the filter house with a wrench. Start at the south end of the filter and open this screw-down rod one complete turn. Move clockwise around the top and open each rod one turn. Continue this until the filter hold-down frame clears the filter frame.

j. Insert the push rod into the east bank push-rod opening located on the north side of the filter house.

k. Slowly push the rod into the house; this pushes the filter bank into the filter box.

NOTE: Health Physics personnel will survey the box for radiation levels during ejection of the filter into the box.

l. Lower and lock the box lid into place.

m. Remove the plastic from the house face. Tape the end of plastic.

n. Transfer the wooden filter shield box to the hot truck.

NOTE: Supervision will prepare burial ground transfer papers and arrange for transfer of the wooden box to the burial ground. Supervision will inform the burial ground supervisor to bury the filter immediately.

2. Center Bank CWS Filters

a. Repeat Steps B.1.b through B.1.n above if the center bank of CWS filters is to be changed.

3. West Bank CWS Filters

a. Repeat Steps B.1.b through B.1.n above if the west bank of CWS filters is to be changed.

NOTE: On Steps B.1 and B.2 above, substitute center and west, respectively, for the word east.

C. Filter Replacement

In the decontamination and cleanup of the filter ways, supervision will personally inspect the filter ways and have them cleaned of debris, which will be accomplished by using wipes on long poles. FPDL operators will install the new filters to the satisfaction of the Inspection Engineer, who will DOP test the filters during the installation.

All banks of filters that have been changed out should be reinstalled before holddown rod adjustments are made. These adjustments should be made while the DOP test is in progress.

The operator will adjust cell ventilation as follows:

1. Seal the cell ventilation housing doors with tape and slowly open the cell ventilation housing dampers (supervision will determine which two banks of filters will be put on stream).

NOTE: Supervision will direct what steps should be taken so that a negative pressure of 2.5 inches of water will never be exceeded while the system is being placed on mainstream.

2. Close the bypass filter cell ventilation damper.

3. Readjust the filter house dampers as necessary to obtain a negative pressure in the manipulator cells of between 1.5 and 2.0 inches of water.

4. Remove plastic from the cells and tape from the doors and resume operation.

D. Decontamination and Cleanup

FPDL operators will:

1. Remove all contaminated waste to the yellow Dumpster.

2. Decontaminate and return all tools and equipment to their proper storage areas.

3. Decontaminate the area as necessary and request Health Physics clearance of the contamination zone.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CELL VENTILATION FILTER CHANGE

FILTER PIT

The replacement of the CWS filters in the main filter pit requires a complete shutdown of the FPDL. Supervision will schedule the change-out and issue specific instructions for the performance of the steps of the following procedure. A filter replacement requires full-time supervision by both FPDL and P&E supervisors.

I. Preoperational Check

A. FPDL

FPDL personnel will be responsible for the following:

1. Closing all cells and sealing with tape.
2. Containing the master ends of all the manipulators with plastic bags.
3. Valving off all steam headers.
4. Turning off all in-cell lights.
5. Roping off the cell ventilation pit area.
6. Setting up an entry and exit portal.
7. Providing the following protective clothing and supplies:

Coveralls	Masking tape
Gas masks	Blotting paper
Shoe covers	Rope
Plastic suits	Plastic bags
Gloves	Contamination cans
Air line system	

8. Providing decontamination supplies such as water hose, long tools with attached scrub brushes, liquid soap, etc.

B. Health Physics

Health Physics personnel will be responsible for:

1. Checking CAM's in the building.
2. Supplying dosimeters and film rings .
3. Supplying surveying equipment.

C. P&E

P&E personnel will be responsible for the following:

1. Crane at the FPDL.

2. Hot truck and lowboy at the FPDL.

3. Special CWS steel filter shield.

4. Special wooden boxes and slide drawers.

5. Special hooks, rods, plastic bags, rope, bolts, shackles and stakes, 3-foot 7/8-inch T socket wrench, and long-handled tongs.

II. Operation (FPDL Personnel)

A. Bypass Filter Station

1. Open the cell ventilation bypass filter damper.

2. Slowly close the cell ventilation house dampers.

NOTE: Supervision must direct the necessary in-building action, such as placing plastic over the cell inlet filters, bagging of manipulators, etc., in order to maintain a minimum negative pressure of 0.25 inch of water in the cells while the building is on the bypass system.

B. Filter Removal

1. East Bank CWS Filters

a. Lay blotter paper and plastic on top of the filter pit and on the surrounding ground.

b. Remove the lead shield from the north manhole cover.

c. Remove the manhole cover and wrap it in plastic. Set cover to one side.

d. Remove the block over the east bank of CWS filters, set on plastic sheet, wrap, and seal the plastic with tape. Put the block to one side.

e. Release the filter locking cams with the 3-foot 7/8-inch T socket wrench.

f. Move the steel filter removal shield (this shield should have the wooden box previously inserted into the shield) into position over the east filter opening.

g. Connect the long-handled rods through the top of the filter shield to filter.

h. Use the crane and slowly pull the bank of filters into the filter shield.

NOTE: Health Physics should monitor this operation closely, noting the personnel exposure and the radiation level on the surface of the shield.

i. Lock the filter into the shield with the specially prepared locking system.

j. Place the wooden cover into the bottom of the box.

k. Release the crane from the filter.

l. Connect the crane to the steel filter shield.

m. Move the filter shield over to the area where plastic sheet has been prepared.

n. Wrap shield in the plastic, tape the plastic, and move the filter shield to the lowboy.

o. Shackle the shield to the lowboy.

p. Prepare burial ground transfer papers.

2. Filter Disposal

P&E personnel will be responsible for disposal of filters as follows:

a. Transfer the shield to the burial ground via Gate 7.

b. Remove the shield from the lowboy at the burial ground and lower into the trench.

c. Release the holding rods and remove the shield from the filter.

d. Cover the filters with fresh earth at the burial ground.

NOTE: The filter shield will be checked by Health Physics personnel at the burial ground and flushed with water if necessary.

P&E personnel will return the shield to the FPDL.

3. West Bank CWS Filters

NOTE: If radiation is low over the east bank of filters and the radiation level at the filter shield is less than 2 r/hr, the west bank of filters may be removed into the same shield containing the east bank of filters and be transferred to the burial ground with the east bank of filters.

a. Remove the block over the west bank of CWS filters, set on plastic sheet, wrap, and seal the plastic with tape. Set the block to one side.

b. Repeat Steps B.1.a through B.1.p and Steps B.2.a through B.2.d.

C. Filter Pit Decontamination

Supervision will personally inspect the pit for condition of the filter framework, north side Neva-clog spray nozzles, condition of paint, etc. He will direct any necessary decontamination efforts to be taken at this time.

D. Filter Replacement

1. P&E personnel will install new filters to the satisfaction of the Inspection Engineer as follows:

a. The gasket will be inspected from time to time to assure that it is intact during installation of the filter bank.

b. The gaskets will be sealed securely against the filter frame.

c. The blocks, manhole covers, and manhole cover shields will be replaced.

d. The blocks will be sealed with tar.

2. FPDL operator will adjust cell ventilation as follows:

a. Valve-in the dampers slowly at the cell ventilation filter house.

NOTE: Supervision will designate which two banks of filters are to be placed on stream. Supervision will also direct what steps should be taken so that a negative pressure of 2.5 inches of water will never be exceeded while the system is being placed on main stream.

b. Close the bypass filter cell ventilation damper.

c. Readjust the filter house damper as necessary to obtain a negative pressure of between 1.5 and 2.0 inches of water in the manipulator cells.

d. Remove plastic from the cells and resume operation.

3. FPDL operator will then:

a. Remove all contaminated waste to the yellow Dumpster.

b. Decontaminate and return all tools and equipment to the proper storage areas.

c. Decontaminate the area as necessary and request Health Physics clearance of the contamination zone.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CHILLED WATER OPERATION

The closed loop chilled water system at the FPDL serves two functions: It conserves process water by recirculating the vessel coolant water and it provides a closed system to contain radioactivity in the event of a leak between the process solution vessels and the cooling system. Under normal conditions the operation of the chilled water system is nearly automatic. However, because of the importance of this system, any trouble must be checked out immediately.

A. Normal Operation

Check to see that:

1. Makeup tank M1334 is operating at a liquid level of 80 to 90%.
2. Only pump U-734B is operating.
3. The two chiller units are valved in series.
4. Unit No. 2 outlet temperature is 40°C ± 5°C.
5. Demineralized water valve to tank M1334 is open.
6. Holdup tank H209 volume is less than 70%.

B. Operational Corrections

1. If both pumps are running:
   a. Turn off U-734A.
2. If U-734A comes back on:
   a. Contact supervision and begin checking for flow restrictions or unusually high demand on the system.
3. If level in tank M1334 is low or if there is an excessive makeup rate in M1334:
   a. See if demineralized water supply to M1334 is open and check for excess demand on demineralized water flow. If necessary, valve-in process water makeup to M1334 and contact supervision.
   b. Check for loss of chilled water:

(1) Buildup in H209 indicates that chilled water is leaking through a steam condensate valve. Check all vessels that are using chilled water; check solid-storage-well cooling system. Contact supervision.

(2) Buildup in S523 indicates loss of chilled water to a cell and a possible leak in in-cell lines. Check calorimeter lines in Cell 14 and check all manipulator cells. Contact supervision.

(3) No buildup in either H209 or S523 indicates an external leak. Contact supervision.

4. If level in tank M1334 is high:

   a. Check that the automatic makeup valve to M1334 is operating properly.

   b. Check for possible leakage in steam inlet valve to vessels that are using chilled water. Contact supervision.

5. If level in tank H209 (70%) is high:

   a. Turn on pump and pump to 20% liquid level; then turn off pump.

   b. Check H209 liquid level 30 minutes after pumping. If it has increased by as much as 5%, contact supervision.

C. Alarm Action

1. If "high-radiation-chilled-water" alarms:

   a. Immediately contact supervision and Health Physics to check radiation at the chilled water unit.

   b. If radiation is found, supervision will start an emergency shutdown of all operations and begin checking for location of the source of activity.

   c. If the recirculating water loop becomes contaminated, close the discharge valve from the H209 pump to manhole 209 immediately and open the valve from H209 pump to Cell 15.

2. If "high-radiation condensate drain" alarms:

   a. Contact supervision and Health Physics.

b. Turn off steam to all process vessels.

c. Attach "Do Not Operate" warning on H209 pump control.

d. Keep a check on H209 volume. If volume builds up above 70% before the cause of the alarm has been determined:

   (1) Close the discharge valve from H209 pump to manhole 209.

   (2) Open the valve from H209 pump to Cell 15 and pump H209.

e. Pull a sample of the solution from H209 and monitor (or count) it for activity. If this solution is contaminated, supervision will direct a shutdown and start checking for the source of activity.

FISSION PRODUCTS DEVELOPMENT LABORATORY

DEMINERALIZER REGENERATION

To Be Scheduled by Supervision

NOTE: Supervision will check that all process operations requiring demineralized water are to be suspended for at least 16 hours.

A. Cation Tank

Check the cation tank to see that it is filled with water (sight glass No. 1 full). If the water level is low, fill as follows:

1. Close valve "V" (inlet) and close all valves on the panel.

2. Open valves 2 and 5.

3. Open valve "V" slowly and allow the cation tank to fill until water comes out the drain line.

4. Close valves 2 and 5.

5. Open valves 1 and 6 and rinse for 15 minutes.

6. Close valves 1 and 6.

B. Cation Bed

1. Backwash the cation bed as follows:

   a. Open valve 2.

   b. Open valve 5 and adjust flow to between 3 and 4 gallons/minute.

   c. Backwash for 15 minutes.

   d. NOTE: During the backwashing, fines will sometimes be washed out. Observe the drain water in the sight glass and if particles larger than pin-head size are being washed out, reduce the water flow.

   e. Close valves 2 and 5.

2. Regenerate the cation bed as follows:

   a. Open valves 3 and 4 and allow 17 inches of water to flow into the acid container.

   b. Close valves 3 and 4.

c. Add 3.5 gallons (2.5 inches) of HNO_3 to the tank (makeup: 1.5 liters of 70% HNO_3 diluted to 3.5 gallons).

d. Open valves 3 and 6.

e. Open valve 4 slowly. Begin taking hydrometer check samples at sampler "A" until the specific gravity of the solution is 1.026; then maintain valve settings until acid tank is empty.

f. When the acid tank is empty, close valve 4.

g. Allow to rinse for 15 minutes and then close valves 3 and 6.

C. Anion Tank

Check anion tank to see that it is filled with water (sight glass No. 2 full). If the water level is low, fill as follows:

1. Open valve 7.

2. Open valve 9 slowly and allow tank to fill until water comes out of tank to drain.

3. Close valves 7 and 9.

D. Anion Bed

1. Backwash the anion bed as follows:

   a. Open valve 9.

   b. Open valve 7 and adjust to a flow of between 3 and 4 gallons/minute.

   c. Backwash for 15 minutes.

   d. NOTE: During the backwash, fines will sometimes be washed out. Observe the drain water in the sight glass and if particles larger than pin-head size are being washed out, reduce the water flow.

2. Regenerate the anion bed as follows:

   a. Open valves 10 and 11 and add 10 inches of water to the caustic tank.

   b. Close valve 10.

   c. Add 10 pounds of NaOH to the water in the caustic tank and mix thoroughly.

   d. Open valve 12.

e. Open valve 10 again, slowly. Begin taking hydrometer check samples at sampler "C" until the specific gravity of the solution is 1.076; then maintain valve settings until caustic tank is empty.

f. When the caustic tank is empty, close valve 10.

g. Allow the bed to rinse for 15 minutes and then close valves 11 and 12.

E. Combined Service Rinse

1. Check that valves "V" and 1 are open.

2. Open valves 8 and 12 to a service rinse flow of between 7 and 8 gallons/minute.

3. Measure the grains per gallon as follows:

   a. Turn switch to on.

   b. Measure the room temperature with a thermometer and set the instrument temperature dial to that temperature.

   c. Adjust the "grains per gallon" dial until the electric eye indicator is a uniform green color.

4. Allow unit to rinse until the "grains per gallon" measurement is below 10; then rinse for an additional hour.

5. Close valve 12.

6. Open valve 13 (service outlet) and adjust flow to between 7 and 8 gallons/minute.

7. Open valve 4 and run 10 gallons of water into the acid tank; discard this water to waste.

8. Open valve 10 and run 10 gallons of water into the caustic tank; discard this water to waste.

9. Check the "grains per gallon" measurement every 2 hours until two consecutive readings give the same measurement ±2; then report the final measurement to supervison (if above 10, the unit will need to be regenerated again).

FISSION PRODUCTS DEVELOPMENT LABORATORY

CHEMICAL MAKEUPS

A. Protective Equipment

NOTE: All operating personnel will wear the following protective equipment during chemical makeup:

1. Face shields.
2. Rubber gloves.
3. Coveralls with sleeves fastened at the wrist.
4. Safety shoes.
5. Respirators while handling dry chemicals.

B. Operating Procedure

1. Pre-makeup checks:

   a. Empty and flush makeup tank with water.

   b. Add required quantity of water to the tank and cool to a temperature of 20°C.

   c. Turn on cooling water on the tank coils.

   d. Turn on mechanical agitator and see that it is giving the proper agitation to the solution.

   e. See if tank ventilation system is on and if the solids-addition lids to all other makeup tanks are closed. Check for a definite air sweep into the makeup tank being used.

   f. Turn off water addition valve to prevent overflowing while adding chemicals.

2. Makeup:

   a. Add all dry chemicals with a scoop or shovel and hold tank temperature below 50°C.

   b. Add acid solutions very carefully while holding tank temperature below 50°C. Acids may be added via Vanton pump, stainless steel bucket, or glass bottles.

FISSION PRODUCTS DEVELOPMENT LABORATORY

GENERAL DECONTAMINATION

A. Cell Equipment Decontamination

1. Secure all jet switches going to tanks that contain radioactive solutions. If additional safety is required, request I&C personnel to disconnect the air to the switch.

2. Check with building supervisor on the path of the decontamination solutions. Write up procedure for moving solution from vessel to vessel.

3. Add the following solutions, in the order listed, to the starting vessels, heat to 90°C for 15 minutes, cool, and jet to the next tank.

   a. 200 gallons water.

   b. 200 gallons 4 N HNO_3 acid.

   c. 200 gallons water.

   d. 200 gallons 4 M NaOH.

   e. 200 gallons water.

   f. 200 gallons 0.1 M sodium versenate.

   g. 200 gallons water.

   h. 200 gallons 0.5 M NaOH - 0.01 M $KmNO_4$.

   i. 200 gallons water.

   j. 200 gallons 0.05 N HNO_3 plus 30 pounds acid.

   k. 200 gallons water.

4. Add 200 gallons of water to each vessel and carefully heat to 100°C. Turn off condenser water and allow steam to decontaminate off-gas system. Control off-gas to not less than 3 inches.

5. Remove cell blocks and have Health Physics check for high-level radiation points. Note these lines or vessels and repeat chemical treatment through these areas.

6. Spray cells and equipment with the following:

a. Hot water.

b. 0.1 M Na versenate.

c. Hot water.

d. 1 M NaOH.

e. Hot water.

f. 1 N HNO_3.

g. Hot water.

7. Keep sprays at a distance from centrifuge motors. Do not operate agitators or centrifuges while spraying.

FISSION PRODUCTS DEVELOPMENT LABORATORY

ELUTION OF ^{137}Cs FROM STT's

NOTE: This procedure applies to both the regular Model II STT and the Model III "gun barrel." Points at which the steps differ are noted in the procedure.

A. Preoperational Checks

1. Enter Cell 15 under cell entry procedures and check that the following valves are closed:

   a. Cell 15 to W19 and W20.

   b. Cell 15 to off-gas.

   c. Vacuum to STT.

   d. R11 to Cell 15.

   e. Cell 15 to E16, P45, and P12.

2. Check that all valves on the Cell 1 panelboard are closed.

3. Check with supervision for approval to open Cell 1N.

4. Remove the blocks from Cell 1N, enter Cell 1N under cell entry procedures, and check that the following valves are closed:

   a. Cell 1 to off-gas.

   b. HAPO to J56-1.

   c. HAPO to J132-1.

   d. Feed to HAPO.

   e. Feed to STT.

   f. STT to J56-1.

5. Open the Cell 1N cell ventilation damper.

6. Check the Cell 1N T.V. camera tilt, pan, and focus.

7. Check that the Snaptite connectors and the lines on the following are in good condition (Model-III lines have HAPO connectors):

   a. Feed to STT (for Model III, feed to HAPO).

b. STT to J56-1 (for Model III, HAPO to J56-1).

c. STT to off-gas (for Model III, HAPO to off-gas).

d. STT test line (not required for Model III).

e. Liquid level probes (2).

8. Set the above connectors in the storage rack.

9. Open the following manual valves in Cell 1N:

a. Feed to STT (for Model III, feed to HAPO).

b. STT to J56-1 (for Model III, HAPO to J56-1).

10. Evacuate Cell 1N but do not replace blocks.

11. Check that the following tanks are clean and empty (except for ^{137}Cs solution from a previous elution):

a. P12, H12, and F12.

b. W19, W20, and S122.

c. P45 and H45.

d. M131, M231, and R11.

12. Check that the following tools are clean and in good repair:

a. Snaptite connector handling tool (T-bar) (HAPO T-bar for Model III).

b. Special tool for adjusting STT long leg (not required for Model III).

c. Standard long tongs and hooks.

d. U-bar for remote opening of manual valves.

e. Small adapter hooks for gasket handling.

f. Long socket wrench for cap removal.

g. Special tools for Model III as follows:

(1) Long Allen wrench (5/8 inch hexagonal) for process line caps.

(2) Impact wrench with 5/8-inch adapter.

(3) Lift tool for caps.

(4) Long Allen wrench (1/2 inch hexagonal) for HAPO connectors.

(5) HAPO connector handling tool.

13. Make up 5 M NH_4NO_3 in M131 and M231. For each 8 gallons of 5 M NH_4NO_3 use 5 gallons of NH_4NO_3 solution from the storage tank (or truck) and 3 gallons of process water.

14. Check that an adequate supply of gaskets is available.

B. Cask Handling

NOTE: When STT's are received they will be monitored by Health Physics and stored as directed by FPDL supervision. Supervision will direct the sequence of elutions.

1. Remove the fire shield retaining rings and bolts from the STT and store them as directed by supervision.

2. Remove the spider from the STT; store the spider and bolts with the fire shield rings. Mark the fire shield rings with the STT number for storage.

3. Remove the nuts from the cupola cover (use impact wrench) and store them with the other items (Steps 1 and 2).

4. Wrap the STT in plastic up to the edge of the cupola.

5. Insert the STT into Cell 1N and position it as instructed by supervision. Remove chains and lugs.

6. Remove the cupola cover from the STT, using the crane. Lift the cover out of the cell, survey, wrap in plastic, and store.

7. Enter Cell 1N under cell entry procedures and connect the thermal liquid level probe. Remove the cap from the shell test nozzle and connect the "STT pressure test" line to the shell test nozzle (shell test connections not required for Model III).

8. Evacuate Cell 1N; insert the armor plate shield, the plastic shield, and the safety railing.

NOTE: The area at the top of Cell 1N will be designated a Danger Zone and the following regulations will apply:

a. Work permit required.

b. Coveralls, safety shoes, gas mask.

c. Two dosimeters, film badge, pencil meters.

d. Portable survey equipment (cutie pies and GM meters).

e. Contamination zone regulations will be applied.

9. Set up a contamination zone adjacent to the work area for storage of tools; put blotter paper down to receive tools. Set up a supply of plastic bags and tape for wrapping.

10. Move the crane to the east end of the crane bay.

11. Check with supervision for approval to begin the cask connection.

12. NOTE: Use Section C for Model II; use Section D for Model III.

C. Cask Connection for Model II

NOTE: Long tools will be used to do the following operations. The tools will be inserted and withdrawn through openings in the plastic shield. Once a tool has entered the danger zone it will be kept in the zone or stored in the designated contamination area until cleared for removal. Tools and equipment being removed from the cell will be monitored and handled according to standard procedures.

1. Insert the wrench and remove the nuts from the vent leg cap.

2. Remove the cap from the vent leg; lift it out of the cell, survey, and wrap in plastic. Store the cap (with nuts).

3. Attach the Snaptite connector handling tool to the connector on the "STT to off-gas" line. Set the Snaptite connector on the STT vent nozzle and connect it by pulling back the collar (use a long hook in the ring) and releasing the collar so that the Snaptite engages the nipple. Test the connection by pulling straight up on the tool. When the connection is firm, remove the tools from the cell.

4. Close the plastic shield.

5. On the Cell 1 panelboard, open the valve from the STT to off-gas. Read the pressure on PI-2019 in the cell and record it on the data sheet.

6. Using the U-bar, open the manual valve on the STT to the off-gas line and vent the cask. Leave both valves on the off-gas line open while making the rest of the connections.

7. Insert the wrench and remove the nuts from the cap on the fill leg.

8. Remove the fill leg cap from the cell; survey, wrap in plastic, and store the cap (with nuts).

9. Attach the Snaptite connector handling tool to the Snaptite on the "Feed to STT" line. Set the connector on the fill leg nozzle, connect it, and test the connection. Remove the tools.

10. Remove the nuts from the cap on the long leg.

11. Remove the long-leg cap from the cell; survey, wrap in plastic, and store the cap (with nuts).

12. Attach the Snaptite connector handling tool to the Snaptite on the "STT to J56-1" line. Set the connector on the long-leg nozzle, connect it, and test the connection. Remove the tools.

13. Insert the special long-leg adjusting tool and fit it to the nut on the movable section of the long leg. Rotate the movable section of the long leg 45° clockwise, push it down firmly, and rotate it another 45° clockwise to lock the long leg in place. Remove the tool.

14. Remove the armor plate shield, plastic shield, and safety railing from Cell 1N and insert the shielding blocks. Seal the top blocks with tape.

15. Adjust the Cell 1N cell ventilation to -1.5 inches of water.

16. Check with supervision for cleanup instructions for the work area.

17. Check with supervision for approval to begin elution.

D. Cask Connection for Model III

NOTE: The following operations will be done remotely with long tools. The tools will be inserted and withdrawn through openings in the plastic shield. Once a tool has entered the danger zone it will be kept in the zone or stored in the designated contamination area until cleared for removal. Tools and equipment being removed from the cell will be monitored and handled according to standard procedure.

1. Remove the cap from the vent nozzle by the following procedure:

   NOTE: Check with supervision for designation of the nozzle.

   a. Remove the bolts from the outer cap.

   b. Connect the lifting tool to the outer cap, rotate the cap, and lift it from the cell.

   c. Survey the bolts and cap and wrap in plastic.

   d. Remove the gasket, using the long tools.

2. Attach the T-bar handling tool to the connector on the "HAPO to off-gas" line. Set the Snaptite connector on the vent nozzle and connect it by pulling back on the collar (use a long hook in the ring) and releasing the collar so that the Snaptite engages the nipple. Use the tool to push the collar down to locked position. Test the connection by pulling up on the T-bar. When the connection is firm, remove the tools from the cell.

3. Open the cask vent valve with the U-bar. Do not torque open.

4. Open the valve (on the panelboard) on the vent line and record the pressure on STT Data Sheet.

5. Open the manual valve on the Cell 1 off-gas line and vent the cask. Leave the cask under off-gas while making the other connections.

6. Remove the caps from the fill leg and long-leg process nozzles by the following procedure:

   NOTE: Check with supervision for designation of the nozzles.

   a. Remove the bolts from the outer cap.

   b. Connect the lifting tool to the outer cap, rotate the cap, and lift it out of the cell.

   c. Survey the bolts and cap and wrap them in plastic.

   d. Remove the gasket, using long tongs.

7. Remove the blank HAPO connector from the fill-leg process nozzle as follows:

   a. Loosen the connector, using the Allen wrench.

   b. Attach the connector tool to the connector and lift it out of the cell. Survey the connector and wrap in plastic.

   c. Remove the gasket with tongs.

   d. Put a new gasket on the nozzle.

8. Attach the handling tool to the HAPO connector on the line from R11.

9. Set the connector (R11 line) on the fill-leg nozzle. Insert the Allen wrench and tighten the HAPO connector. Remove the handling tool.

10. Remove the blank HAPO connector from the long-leg process nozzle as follows:

a. Loosen the connector, using the Allen wrench.

b. Attach the lifting tool to the connector and lift it out of the cell. Survey the cap and wrap in plastic.

c. Remove the gasket with tongs.

d. Put a new gasket on the nozzle.

11. Attach the handling tool to the HAPO connector on the line to J56-1.

12. Set the connector (J56-1 line) on the long-leg nozzle. Insert the Allen wrench and tighten the HAPO connector. Remove the handling tool.

13. Close all openings in the plastic shield. Isolate the work area over Cell 1N. No entry will be allowed while the connection test is being made.

14. Check that all valves on the Cell 1 panelboard are closed.

15. At the panelboard:

a. Open off-gas to the cask.

b. Open the valve from the J56-1 line to ____ only (check with supervision).

c. Open valve from cask to J56-1.

d. Turn on water to D51.

16. Turn on the jet and observe the cask probe lights.

17. Operate the jet until a red probe light is obtained. Close the valve from the cask to J56-1. Shut off J56-1. Close the valve from J56-1 to _____.

18. Open the valve from the funnel to the cask on the panelboard. Add water to the cask at the rate of 2 gallons/minute until a steady green probe light is obtained. Then add 25 more gallons of water.

19. Close the valve from the funnel to the cask on the panelboard. Close the valve to hot off-gas (on the panelboard).

20. Open the valve from R11 to the cask. Open the valve from the cask to J64-1. Open J64-1. Jet to W-_____ until ____ gallons have been transferred. Record the time that jetting was started and was stopped.

21. When the system is leak-tight and operable (check with supervision), remove all tools from the cell. Survey, clean, and wrap in plastic.

22. Check with supervision for cleanup instructions for the work area.

23. Check with supervision for approval to begin elution.

E. Elution Procedure

1. Preheating - Set the controller on the liquid level probe Wheelco at 70°C and check which light is on.

   a. If the red light is on, refill the STT as in Step E.2 below.

   b. If the green probe light is on, proceed to Step E.3.

2. Refilling Procedure - This procedure will be used each time the liquid level in the STT drops below the probe (that is, each time the red light comes on).

   a. Open the valve from STT to off-gas on the panelboard. (for Model III, open "HAPO to off-gas").

   b. Open the valve on the solution addition line to line 1016 on the panelboard.

   c. Add water* to the solution addition funnel at a rate of 3 gallons/minute until 40 gallons has been added; then stop the water addition and close the valve on the solution addition line to line 1016.

      *During NH_4NO_3 elution, use 5 M NH_4NO_3 instead of water.

   d. Close the valve on the STT to off-gas line on the panelboard (for Model III, close "HAPO to off-gas").

3. Add water to R11 until the R11 liquid level reads 95%. Close the valve on STT to off-gas on the panelboard.

4. Turn on the steam to R11 and turn on the R11 air sparger.

5. Heat R11 to 80°C and control at 80°C.

6. Open the following valves:

   a. R11 to STT (2 valves) (for Model III, open "R11 to HAPO").

   b. STT to J56-1 (for Model III, open "HAPO to J56-1").

c. J56-1 to _____(check with supervision).

7. Turn on the steam to heater D61. Set the steam control Wheelco at 80°C.

8. Turn on water to cooler D51.

9. Set the thermal liquid level probe controller at 100°C.

10. Turn on J56-1 and begin jetting to _____.

11. Watch the R11 liquid level and the STT probe light. As R11 liquid level drops, begin adding water to R11 at a rate of 3 gallons/minute. Keep the R11 liquid level between 70 and 95%. If the red probe light comes on:

    a. Close the valve from the STT to J56-1 (for Model III, close "HAPO to J56-1").

    b. Turn off J56-1.

    c. Refill the STT with water as in Step E.2.

12. Continue to operate until a total of 500 gallons of solution has been collected in _____; then:

    a. Close the valve from the STT to J56-1 (for Model III, close "HAPO to J56-1").

    b. Turn off J56-1.

    c. Close the valve from J56-1 to _____.

    d. Close the valve from R11 STT (2 valves) (for Model III, close "R11 to HAPO").

    e. Turn off R11 steam and R11 air sparger.

13. Open R11 to S324 and empty R11; then close the valve.

14. Check with supervision for disposal of the preheat water from _____.

15. Proceed immediately with Section F.

F. Elution Procedure - NH_4NO_3 Elution

1. Heat the 5 M NH_4NO_3 in M131 and M231 to 80°C and control. Add 5 M HN_4NO_3 from M131 or M231 to R11 until the R11 liquid level is 95%.

2. Turn on R11 steam and R11 air sparger. Control R11 temperature at 80°C.

3. Open the following valves:

   a. R11 to STT (2 valves) (for Model III, open "R11 to HAPO).

   b. STT to J56-1 (for Model III, open "HAPO to J56-1).

   c. J56-1 to _____ (check with supervision).

4. Turn on J56-1 and begin elution. Watch the R11 liquid level, the STT probe lights, and the Cell 1N electrometer.

5. As R11 liqud level drops, add 5 M NH_4NO_3 to R11 at 3 gallons/minute. Keep the R11 liquid level between 50 and 90%. If the red probe light comes on:

   a. Close the valve from the STT to J56-1 (for Modell III, close "HAPO to J56-1").

   b. Turn off J56-1.

   c. Refill the STT with 5 M NH_4NO_3 as in Section E.2.

6. Operate until the Cell 1N electrometer shows an increase of as much as 1/2 scale, and then immediately open the valve from J56-1 to S122 and close the valve from J56-1 to _____.

7. Continue to operate, controlling the system as described in Step 4 above.

8. NOTE: Check with supervision for handling of the solution collected in Steps 3 to 6 above.

   As solution from M131 or M231 is used, make up fresh 5 M NH_4NO_3 in these tanks.

9. Operate until _____gallons have been collected in S122 (check with supervision); then open the valve from J56-1 to _____ and close the valve from J56-1 to S122.

10. Operate until _____gallons have been collected in _____ (check with supervision); then:

    a. Close the valve from STT to J56-1 (for Model III, close "HAPO to J56-1).

    b. Turn off J56-1.

    c. Close the valve from J56-1 to _____.

11. Pull a gamma-chamber check sample from _____ and contact supervision.

    Supervision will determine whether the elution is complete or additional elution is necessary.

12. When elution is complete, close the valves from R11 to STT.

13. Turn off R11 steam and R11 air sparger.

14. Open the valve from R11 to S324 and drain R11; then close the valve.

15. Turn off the steam to heater D61.

16. Proceed immediately to Section G.

G. Elution Procedure - Rinsing

1. Add process water to R11 until the R11 liquid level reads 90%.

2. Open the valves from R11 to STT (for Model III, open "R11 to HAPO").

3. Open the valve from the STT to J56-1.

4. Open the valve from J56-1 to _____ (check with supervision).

5. Turn on J56-1 and begin rinsing. Watch the R11 liquid level, and add process water to R11 to maintain the liquid level between 50 and 95%. If the red probe light comes on:

   a. Close the valve from the STT to J56-1 (for Model III, close "HAPO to J56-1").

   b. Turn off J56-1.

   c. Close valve from J56-1 to _____.

   d. Refill STT as in Section E.2.

6. Operate until the collection tank reaches 90%; then:

   a. Close the valve from the STT to J56-1 (for Model III, close "HAPO to J56-1").

   b. Turn off J56-1.

   c. Close the valve from J56-1 to _____.

7. Sample _____(Code _____Cs2-S1) and jet to waste.

8. Repeat Steps 3 to 7 above until a total of 1500 gallons of rinse water has been discarded. Sample each batch (Codes _____Cs2-S2, _____Cs2-S3, etc.). Composite the samples at 1 milliliter per 50 gallons and submit to Analytical.

9. Close all valves on the Cell 1 panelboard.

10. Open the valve from the STT to off-gas (on the panelboard).

11. NOTE: Use Section H for Model II; use Section I for Model III.

H. Cask Disconnect for Model II

1. Check with supervision for approval to open Cell 1N.

2. With constant Health Physics survey, remove the shielding blocks from Cell 1N; insert the armor plate shield, plastic shield, and safety railing.

3. Set up the work area over Cell 1N as described in Section B, beginning with Note under Step 8 and going through Step 12.

4. Insert the long-leg adjusting tool and fit it to the nut on the movable section of the long leg. Rotate the leg 45° counterclockwise, lift the leg, then rotate it another 45° counterclockwise to lock the leg in the vent position. Remove the tool.

5. Insert the Snaptite connector handling tool and attach it to the Snaptite on the line from the STT long leg to J56-1. Insert a hook and engage it in the ring on the Snaptite collar. Pull the collar back and disconnect the Snaptite. Set the Snaptite and line in the storage rack and disengage the tools.

6. Attach the Snaptite handling tool to the Snaptite on the line from R11 to the STT. Pull the collar back and disconnect the Snaptite. Set the Snaptite and line in the storage rack. Remove the tools from the cell.

7. Remove all used gaskets from the STT cupola.

8. Insert a new gasket on the flange for the long-leg cap. Insert the cap and position it over the long-leg nozzle. Insert the bolts into the cap and tighten them evenly. Finally tighten all bolts with maximum torque from the long manual wrench.

9. Place the cap on the short-leg nozzle by the same operation as in Step 8.

10. Check with supervision for test procedure instructions; if pressure test is to be made, make it at this point according to Section J below.

11. Next, insert the Snaptite connector handling tool and attach it to the Snaptite on the STT vent line. Pull the Snaptite collar back with a long hook and disconnect the Snaptite. Set the Snaptite and line in the storage rack and remove the tool.

12. Insert a new gasket on the flange for the vent cap. Insert the vent cap and position it over the vent nozzle. Insert the bolts into the vent cap and tighten them evenly. Finally tighten the bolts with the maximum torque with the long manual wrench. Close the valve from STT to off-gas (on the panelboard).

13. Check with supervision for approval to enter Cell 1N. Supervision will determine whether remote decontamination is necessary.

14. Enter Cell 1N under cell entry procedures and disconnect the probe. Disconnect the STT shell test line and cap the nozzle.

15. Close the following manual valves:

    a. Feed to STT.

    b. STT to J56-1.

    c. STT to off-gas.

16. Decontaminate the inside of the cupola to a direct reading of less than 100 mr/hr and a smear reading of less than 1 mr/hr on a standard smear.

17. Insert the gasket into the cupola. Insert the cupola cover and set it on the cupola. Attach the nuts and tighten them manually. Finally tighten the nuts with the impact wrench.

18. Remove the plastic from the STT and smear. Decontaminate to less than 1-mr/hr smear level.

19. Remove the armor plate and plastic shields.

20. Remove the STT from Cell 1N and decontaminate it to shipping tolerance.

21. Attach the spider assembly to the cupola.

22. Replace the fire shield retaining rings and bolts.

23. Check with supervision for Cell 1N cleanup instructions.

24. Unless another STT is to be inserted immediately, replace the shielding blocks in Cell 1N.

25. Check with supervision for removal of Danger Zone regulations.

I. Cask Disconnect for Model III

1. Check with supervision for approval to open Cell 1N.

2. With constant Health Physics survey, remove the shielding blocks from Cell 1N; insert the armor plate shield, plastic shield, and safety railing.

3. Set up the work area over Cell 1N as described in Section B, beginning with Note under Step 8 and going through Step 12.

4. Attach the handling tool to the HAPO connector on the line to the J56-1 jet (this line is on the long-leg nozzle). Loosen the connector, remove it from the nozzle, and set the connector in the storage rack. Remove the handling tool.

5. Attach the handling tool to the HAPO connector on the line from R11 (this line is now on the feed nozzle). Loosen the connector, remove it from the nozzle, and set the connector in the storage rack. Remove the handling tool.

6. Remove all gaskets from the cupola.

7. Insert new gaskets onto the long-leg and feed nozzles.

8. Set the blank connectors onto the long-leg and feed nozzles and tighten them down as well as possible with the Allen wrench.

9. Place the two outer caps over the blank HAPO connectors as follows:

   a. Insert a new O-ring gasket.

   b. Attach the outer cap to the handling tool and check that the hold-down bolts are in place.

   c. Put the cap on the process nozzle (over the HAPO connector).

   d. Turn the hold-down bolts with the long wrench until all are started in the threads. Begin tightening the bolts a few turns at a time; proceed from one bolt to the next and tighten them evenly. Manually tighten all bolts as firmly as possible.

10. When both outer caps have been tightened, perform the pressure test as follows:

    a. Insert a hose into the cupola and add water to a point just over the outer cap bolts. Careful! DO NOT FILL THE CUPOLA.

b. Close the manual valve on the line from cask to off-gas. Leave the cask vent valve and the cask to off-gas valve on the panelboard open.

c. Open the two valves on the air pressure to the vent system (these are designated HX-20111 and HX-20104) and set the pressure regulator (PIC-20111) at 90%.

d. Read the pressure on the vent line (PI-2019).

e. Observe the water in the cupola for air bubbles and watch the pressure gage for any decrease. If either of these is observed, contact supervision.

f. After caps have been determined to be leak-tight or the leaking caps have been identified, open the manual valve on the line from cask to off-gas.

g. Set the air pressure regulator (PIC-2011) at zero.

h. Open the two valves on the air pressure to the vent system (HX-20111 and HX-20104). Leave these valves open for 10 seconds, then close them.

i. Attach the Snaptite suction line to the line from Cell 1N to P12. Jet the water from the cupola to P12, then turn off the jet.

j. Dry the cupola by swabbing. Place the swabs in a container in Cell 1N.

k. If any leaks are found, the caps must be made leak-tight before proceeding. Check with supervision.

l. When system is leak-tight, proceed with final disconnect of the cask.

m. Record final leak test pressure on STT Data Sheet.

11. Close the following valves:

a. Cask vent valve.

b. Manual valve on line from cask to off-gas.

c. Valve from cask to off-gas on panelboard.

12. Attach the T-bar tool to the Snaptite on the cask vent nipple and remove it from the cask. Set the line in the storage rack.

13. Insert the outer cap over the off-gas nozzle as follows:

a. Insert a new O-ring gasket.

b. Attach the outer cap to the handling tool and check that the hold-down bolts are in place.

c. Insert the cap onto the nozzle.

d. Turn the hold-down bolts with the long wrench until all are started in the threads. Begin tightening the bolts a few turns at a time; proceed from one bolt to the next and tighten them evenly. Manually tighten all bolts as firmly as possible.

14. Check with supervision for approval to enter Cell 1N. Supervision will determine whether remote decontamination is necessary.

15. Enter Cell 1N under cell entry procedures and disconnect the probes. Install new gaskets around the probes and install the probe caps. Tighten the caps uniformly.

16. Close the following manual valves:

a. Feed to HAPO.

b. HAPO to J56-1.

c. HAPO to off-gas.

17. Decontaminate the cupola of the cask to a probe reading of less than 1 mr/hr on a standard smear.

18. Insert the gasket onto the cupola. Insert the cupola cover and set it on the cupola. Attach the nuts and tighten them manually. Finally tighten the nuts with an impact wrench.

19. Remove the plastic from the cask and smear. Decontaminate to less than 1-mr/hr smear level.

20. Remove the armor plate and plastic shields.

21. Remove the cask from Cell 1N and decontaminate it to shipping tolerance.

22. Record final smear results, survey results, and shipping date. Supervision and Health Physics will certify the cask as ready for shipment.

23. Turn in all Data Sheets for filing.

24. Check with supervision for Cell 1 cleanup instructions.

25. Unless another cask is to be inserted immediately, replace the shielding blocks in Cell 1N.

26. Check with supervision for removal of Danger Zone regulations.

J. Pressure Tests for STT Model II

1. Cavity Test

a. Close valve HX-20104 on Cell 1 panelboard.

b. Fill the STT cupola with water.

c. Open valve HX-20111; then adjust pressure on PI-2011 to 50 psig, using the controller.

d. Hold the 50-psig pressure for 30 minutes and watch for bubbles in the cupola water. If bubbles are observed, contact supervision.

e. If no bubbles have been observed after 30 minutes, close valve HX-20111 and set pressure controller to zero.

f. Open valve HX-20112 and leave open for 5 minutes; then close HX-20112 and open HX-20104.

g. Leave water in cupola.

2. Shell Test

a. Check that pressure test line is connected.

b. Open HX-20106, check pressure on PI-20106. The pressure should be 15 ± 1 psig; if not, contact supervision.

c. Close HX-20106. Watch PI-20106 for pressure drop. If pressure drops, look for bubbles in the cupola and identify their location; then contact supervision.

d. If there is no pressure drop after 30 minutes, disconnect the shell test line.

e. Vent the shell by depressing the Snaptite valve stem with the long tool.

f. Contact supervision for instructions on removal of water from cupola.

FISSION PRODUCTS DEVELOPMENT LABORATORY
STT ELUTION DATA SHEET

Run No._____

STT No._____ Date Received_____

Date Vented_____ Pressure_____psig

Pre-heat, Elution, Rinse*
(Record time and date)

Process line connections completed_____ Supr._____

Preheat: Started_____ Completed_____

Elution: Started_____ Completed_____

Rinse: Started_____ Completed_____

Disconnect completed_____ Supr._____

Pressure test completed_____ Supr._____

Decontamination completed_____ Supr._____

*Record data below, use data sheet 2 if required

Time/date	Solution	Cell 1N Electrometer	Receiver tank	Vol. in receiver	Remarks

Supervisor_____

STT ELUTION DATA SHEET #2 Run No._____

Time/date	Solution	Cell 1N Electrometer	Receiver tank	Vol. in receiver	Remarks

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 FEED PREPARATION

A. Operational Checks

1. Check that all valves and jets on the Cell 5 panelboard are closed.

2. Check with supervision for designation of the feed storage tank from which to transfer feed.

3. Check that the valves from the feed storage tank to P45 are set properly.

   a. For transfer from S122 to P45:

      (1) Check that the valve from S122 to P12 is closed.

      (2) Open the valve from S122 to P45.

   b. For transfers from W19 or W20 to P45:

      (1) Check that the valves from jet J57-25 to E16 and C17 are closed.

      (2) Open the discharge valve from J57-25 and open the valve from J57-25 to P45.

      (3) Open the intake valve to J57-25.

      (4) Open the appropriate manual valve. These valves are labeled "W19 to U125" and "W20 to U125"; close the other manual valve.

4. Check that P45 is empty.

B. Operation

1. Turn on the chilled water to P45 jacket.

2. Turn on the jet from the feed storage tank to P45 and jet_____ gallons of solution to P45; then turn off the jet.

3. Turn on the P45 agitator.

4. Slowly add_____gallons of 60% HNO_3 to P45. Control P45 temperature below 50°C.

NOTE: The amount of 60% HNO_3 to be added is 5 gallons for each 100 gallons of feed in P45.

5. Turn off the chilled water to P45 jacket.

6. Turn on steam to P45 jacket and heat P45 to_____°C.

7. Digest P45 at_____°C for_____minutes.

8. Turn off steam, and turn on chilled water to P45 jacket.

9. Cool P45 to 35°C, and then turn off chilled water.

10. Pull a 10-milliliter sample of P45 solution. Code_____Cs7-C _____. Inspect the sample for precipitate and if any precipitate is present, contact supervision.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 FIRST CRYSTALLIZATION

A. Operational Checks

1. Check that the feed preparation operation in P-45 is complete.

2. Check that C-17 is empty except for crystal bed. (Unless this is the first batch after a half-batch, transfer from C-17 to C-38; see Section D.1 below).

3. Check that all valves and jets to and from C-17 are closed.

4. Make out a Batch Data Sheet for this run and fill in the "Status in C-17" section.

B. Feed Crystallization

1. Open the valve and turn on the jet from P-45 to C-17.

2. Jet _____ gallons of solution from P-45 to C-17; then close the valve and turn off the jet.

3. NOTE: If this batch transfer will empty P-45, turn off the P-45 agitator when the P-45 liquid level reaches 15%. When P-45 is empty, add 10 gallons of water to P-45, jet it to C-17, and then turn off the jet.

4. Add enough water to C-17 to bring the liquid level up to __%.

5. Turn on the C-17 agitator and the contact mike.

6. If the previous crystal bed in C-17 was less than ___ gallons, add _____ pounds of alum to C-17 through the solids addition funnel; otherwise, contact supervision.

7. Turn on steam to C-17 and heat C-17 to _____ °C.

8. Digest C-17 at _____°C for _____ minutes.

9. Check with supervision if gamma check sample is to be taken at this point.

10. Turn off steam to C-17.

11. Turn on the air to the C-17 filter blowdown.

12. Turn on chilled water to C-17 and cool until a temperature break is noted.

13. Record temperature break and crystal volume.

14. Continue cooling until the C-17 temperature is _____°C.

15. Continue agitating C-17 for _____ minutes from the time of the temperature break. Control C-17 temperature at ____°C.

16. Turn off the C-17 agitator and turn off the filter blowdown air.

17. Allow the C-17 crystal bed to settle for _____ minutes.

18. Check that C-27 is empty except for crystal bed.

19. Check that all valves and jets to and from C-27 are closed.

20. Turn on the jet from the C-17 decant leg to C-27 and decant the solution; when the C-17 liquid level reaches _____%, turn off the decant jet.

21. Open the valve from the C-17 filter jet to C-27 and turn on the jet.

22. Filter the remaining solution from C-17 to C-27.

23. NOTE: If the filter jet stops pulling solution before C-17 is empty (as indicated by the jet thermocouple or by the C-17 liquid level), turn off the jet, close the valve, and turn on the filter blowdown air for 1 minute; then turn off the filter blowdown air, open the valve, and turn on the jet.

    Control C-27 temperature below 60°C.

24. When the filtration is complete, turn off the filter jet and close the valve from C-17 to C-27.

25. Turn off chilled water to C-17.

26. Proceed with scavenge crystallization in C-27.

C. Water Crystallization

NOTE: Check with supervision before starting a water crystallization

1. Add _____ gallons water to C-17.

2. Turn on the C-17 agitator and the contact mike.

3. Turn on steam to C-17 and heat C-17 to 90°C.

4. Digest C-17 at 90°C for 15 minutes.

5. Turn off steam, turn on chilled water to C-17, and cool until a temperature break is noted.

6. Record temperature break and crystal volume.

7. Continue cooling until the C-17 temperature is _____°C.

8. Continue agitating C-17 for _____ minutes from the time of the temperature break. Control C-17 temperature at ___°C.

9. Turn off C-17 agitator and turn off the filter blowdown air.

10. Allow the C-17 crystal bed to settle for _____ minutes.

11. Check that C-27 is empty except for crystal bed.

12. Check that all valves and jets to and from C-27 are closed.

13. Turn on the jet from the C-17 decant leg and decant the solution. When the C-17 liquid level reaches _____%, turn off the decant jet.

14. Open the valve from the C-17 filter jet to C-27 and turn on the jet.

15. Filter the remaining solution from C-17 to C-27.

16. NOTE: If the filter jet stops pulling solution before C-17 is empty (as indicated by the jet thermocouple or by the C-17 liquid level), turn off the jet, close the valve, and turn on the filter blowdown air for 1 minute; then turn off the filter blowdown air, open the valve, and turn on the jet.

    Control C-27 temperature below 60°C.

17. When the filtration is complete, turn off the filter jet and close the valve from C-17 to C-27.

18. Turn off the chilled water to C-17.

19. Proceed with scavenge crystallization in C-27.

D. Cesium Transfers to C-38

NOTE: This operation will be done when the ^{137}Cs content in C-17 gets above _____ curies, or as otherwise directed by supervision. Supervision will determine whether half-batch or full-batch transfer is to be made.

1. Half-Batch Transfer

   a. Run a sufficient number of water crystallizations (as in Section C above) to reduce the crystal bed in C-17 to less than 10 gallons.

   b. Add 150 gallons of water to C-17.

   c. Turn on the C-17 agitator and the contact mike.

   d. Turn on the steam to C-17 and heat C-17 to 90°C.

   e. Digest C-17 at 90°C for 1 hour.

   f. Turn off the steam to C-17. Turn on the chilled water to C-17 and cool to 50°C; control C-17 at 50°C.

   g. Check that C-38 has no more than 25 gallons of solution in it. Check with supervision for approval to make the transfer.

   h. NOTE: If the transfer to C-38 cannot be made, transfer to E-16 for temporary storage. Check that E-16 has at least 100 gallons free volume.

   i. Open the valve and turn on the jet from C-17 to C-38 (or E-16). Open the valve from C-17 to C-38 (or E-16) and jet 75 gallons of solution from C-17 to C-38 (or E-16); then turn off the jet and close the valve.

   j. NOTE: Control C-38 (or E-16) temperature below 60°C.

   k. Turn off the C-17 agitator.

   l. Contact supervision for crystallization instructions on C-38.

   m. Resume crystallization operations in C-17.

   n. NOTE: On the first batch in C-17 after a half-batch transfer from C-17 to C-38, there will be 75 gallons of solution remaining in C-17. When making out the Batch Data Sheet, assume that 50% of the ^{137}Cs content in C-17 was transferred to C-38 <u>or</u> determine ^{137}Cs by gamma check sampling.

2. Full-Batch Transfer

   a. Run a sufficient number of water crystallizations in C-17 to reduce the crystal bed to less than 10 gallons.

b. Add _____ gallons of water to C-17.

c. Turn on the C-17 agitator and the contact mike.

d. Turn on the steam to C-17 and heat C-17 to 90°C.

e. Digest C-17 to 90°C for 1 hour.

f. Turn off the steam to C-17 and turn on the cooling water. Cool C-17 to 50°C and control at 50°C.

g. Check that C-38 is empty except for crystal bed.

h. Check with supervision for approval to make the transfer to C-38.

i. NOTE: If the transfer to C-38 cannot be made, transfer to E-16 for temporary storage. Check that E-16 has at least 150 gallons of free volume.

j. Open the valve and turn on the jet from C-17 to C-38 (or E-16).

k. When the C-17 liquid level reaches 10%, turn off C-17 agitator.

l. NOTE: Control C-38 temperature below 60°C.

m. When C-17 is empty, turn off the jet. Add 10 gallons of water to C-17 and immediately jet it to C-38 (or E-16); then turn off the jet and close the valve.

n. Check with supervision for crystallization instructions for C-38.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 CRYSTALLIZATION

DATA SHEET

Date/time started feed transfer_____

Batch number_____ Volume feed_____ Feed from_____

C-17 OPERATION

In C-17 before batch:_____ curies_____ gal crystals

Additions to C-17:_____ gal acid_____ gal water_____ lb alum

In C-17 before crystallization_____ gallons_____ curies

Crystallization Cycle: initial temp.____ °C (record times and temp. below).

Started heating_____ Reached digestion temp._____ (____ °C)

Started cooling_____ Reached crystal break_____ (____ °C-_____ gal cryst)

Started settling_____ (@____ °C) Started filtration_____ Completed fil_____

C-27 OPERATION

In C-27 before batch:_____ curies_____ gal crystals

Additions to C-27:_____ lb alum_____ gal water

In C-27 before crystallization:_____ gallons_____ curies

Crystallization Cycle: initial temp.____ °C (record times and temp. below).

Started heating_____ Reached digestion temp._____ (____ °C)

Started cooling_____ Reached crystal break_____ (____ °C-_____ gal cryst)

Started settling_____ (@____ °C) Started filtration_____ Completed fil_____

P-45 OPERATION

In P-45 before batch:_____ curies_____ gal crystals

Additions to P-45:_____ lb alum_____ gal water

In P-45 before crystallization:_____ gallons_____ curies

Crystallization Cycle: initial temp.____ °C (record times and temp. below).

Started heating_____ Reached digestion temp._____ (____ °C)

Started cooling_____ Reached crystal break_____ (____ °C-_____ gal cryst)

Started settling_____ (@____ °C) Started filtration_____ Completed fil_____

Waste vol in H-45_____ gal Gamma check_____ on_____ scale curies_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 SCAVENGE CRYSTALLIZATION

The starting point for this operation is immediately after the filtration of a ^{137}Cs first crystallization or water crystallization batch from C-17 to C-27.

A. Crystallization Operation

1. Turn on the C-27 agitator and the contact mike.

2. Turn on the steam to C-27 and heat C-27 to _____°C.

3. Digest C-27 for 15 minutes at _____°C; then pull a sample from C-27 for gamma check.

4. If the ^{137}Cs content in C-27 is greater than _____ curies, contact supervision (see Step C below).

NOTE: If the ^{137}Cs content in C-27 is within limits, a fresh batch of feed may be started in C-17 at this point.

5. If the previous crystal bed in C-27 was less than _____ gallons, add _____ pounds of alum to C-27.

6. Digest C-27 at _____°C for _____ minutes after alum addition.

7. Turn off steam to C-27.

8. Turn on air to C-27 filter blowdown.

9. Turn on the chilled water to C-27 and cool until a temperature break is noted.

10. Record temperature break and crystal volume.

11. NOTE: If this volume is less than _____gallons, contact supervision.

12. Continue cooling C-27 until the temperature is _____°C.

13. Continue agitating C-27 for _____ minutes after the time of the temperature break. Control C-27 at _____°C.

14. Turn off the C-27 agitator and filter blowdown.

15. Allow the C-27 crystal bed to settle for _____ minutes.

16. Check that S-324 is empty.

17. Open the valve and turn on the jet from C-27 filter leg to S-324.

18. Filter the solution from C-27 to S-324.

19. NOTE: If the filter jet stops pulling solution before C-27 is empty, turn off the jet, close the valve, and turn on the filter blowdown air for 1 minute; then turn off the filter blowdown air, open the valve, and turn on the jet.

20. When C-27 is empty, turn off the jet and close the valve.

21. Turn off the chilled water to C-27.

22. Sample S-324. The sample code will be _____Cs24W_____ if this is a scavenge of a first crystallization batch and will be _____Cs24-S_____ if it is scavenge of a water crystallization batch.

23. Transfer the sample to Cell 18 and make gamma check. If the total ^{137}Cs in S-324 is greater than 100 curies, contact supervision.

24. Composite the sample in the proper composite bottle. Use 1 milliliter per 50 gallons of solution in S-324.

25. Jet S-324 to waste.

B. Bed Transfer to C-17

NOTE: This operation will be done as directed by supervision. Supervision will determine if water crystallizations are necessary in C-27.

1. If water crystallization is needed, add _____ gallons of water to C-27 and then proceed as in Section A above.

2. With C-27 empty except for a crystal bed of _____ gallons, add _____ gallons of water to C-27 (check with supervision).

3. Turn on the C-27 agitator and the contact mike.

4. Turn on the steam to C-27 coil and heat C-27 to 90°C.

5. Digest C-27 at 90°C for 30 minutes; then turn off the steam.

6. Turn on the chilled water to C-27 and cool C-27 to 50°C. Control C-27 temperature at 50°C.

7. NOTE: Watch for a temperature break during the cooling. If a temperature break occurs, turn off the chilled water and contact supervision.

8. Check that C-17 is ready to receive solution.

9. Open the valve from C-27 to C-17 and turn on the jet.

10. Jet the solution from C-27 to C-17. When C-27 liquid level reaches 15%, turn off the C-27 agitator.

11. When C-27 is empty, turn off the jet. Add 10 gallons of water to C-27, jet it to C-17, then turn off the jet and close the valve.

12. Turn off all valves and jets on C-27.

C. Filtrate Return to C-17

NOTE: This operation is to be done when the ^{137}Cs content in C-27 exceeds _____ curies.

1. Turn on the C-27 agitator and the contact mike.

2. Turn on steam to C-27 and heat C-27 to 90°C.

3. Digest C-27 at 90°C for 15 minutes.

4. Turn off steam, turn on chilled water to C-27, and cool C-27 to 50°C.

5. NOTE: Watch for a temperature break during the cooling. If a break is noted, turn off the chilled water and contact supervision.

6. Control C-27 at 50°C.

7. Check that C-17 is empty except for crystal bed.

8. Open the valve from C-27 to C-17 and turn on the jet.

9. Jet the solution from C-27 to C-17. When C-27 liquid level reaches 15%, turn off C-27 agitator.

10. When C-27 is empty, turn off the jet and close the valve.

11. Close all valves and jets on C-27.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 CONCENTRATE CRYSTALLIZATION

NOTE: The starting point for this operation is immediately after a transfer of ^{137}Cs from C17 to C38 or as otherwise instructed by supervision.

A. Crystallization Operation

1. Turn on the C38 agitator and the contact mike.

2. Turn on steam to C38 and heat C38 to 90°C.

3. Digest C38 at 90°C for_____minutes.

4. Turn off steam and turn on the C38 filter blowdown air.

5. Turn on chilled water to C38 and cool C38 to_____°C. If a temperature break is noted, record the crystal volume.

6. Continue to agitate C38 for_____minutes from the temperature break (or from the time that_____°C was reached).

7. Turn off the C38 agitator and turn off the C38 filter blowdown air.

8. Allow the C38 crystal bed to settle for_____minutes.

9. Check that C48 is empty and that all valves and jets to C48 are closed.

10. Open the valve from the C38 filter jet to C48.

11. Turn on the jet and filter the solution from C38 to C48. Control C48 temperature below 60°C.

12. If the filter jet stops pulling solution before C38 is empty (as indicated by the jet thermocouple or by the C38 liquid level), turn off the jet, close the valve, and turn on the filter blowdown air for 1 minute; then turn off the filter blowdown air.

13. When the filtration is complete, turn off the filter jet and close the valve.

14. Turn on the C48 agitator.

15. Turn off the C38 chilled water.

16. Immediately add 25 gallons of water to C38 (unless otherwise instructed by supervision) and turn on chilled water to C38.

17. Turn on the C38 agitator and turn on the filter blowdown air.

18. Check with supervision for handling of the C48 solution.

19. The solution from C48 will normally be jetted back to C17 and combined with the next mainstream feed batch. When this operation is to be done, proceed as follows:

    a. Check that C17 is ready to receive solution.

    b. Determine the ^{137}Cs content in C17 (either by gamma check sample or from previous Batch Data Sheet).

    c. Open the valve from C48 to C17 and turn on the jet.

    d. When the C48 liquid level reaches 15%, turn off the C48 agitator.

    e. When C48 is empty, turn off the jet and close the valve.

    f. Check that all valves and jets to and from C48 are closed.

    g. Determine the ^{137}Cs content in C17 by gamma check.

    h. By difference (Step g minus Step b), determine the amount of ^{137}Cs recycled from C48 and record it on the Batch Data Sheet.

B. Water Crystallization

1. Check with supervision for the volume of water needed for the water crystallization and then add the water to C38.

NOTE: C38 will normally have 25 gallons of solution in it when the operation is started; this must be taken into account when calculating the water addition.

From this point, the water crystallization is identical to the "Crystallization Operation," Section A above.

2. Check with supervision for the number of water crystallizations needed.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 PURIFICATION CRYSTALLIZATION

NOTE: The starting point for this operation is determined by supervision.

1. At the starting time, see that the following conditions exist:

   a. C38 contains 25 gallons of solution.

   b. C38 agitator and filter blowdown air are on.

   c. All jets and valves on Cell 8 panelboard are closed.

   d. C38 is empty.

   e. C38 chilled water is on.

2. Check with supervision for approval to start operation. Supervision will determine whether additional water is to be added to C38.

3. Turn off the C38 chilled water.

4. Turn on steam to C38 and heat C38 to_____°C.

5. Digest C38 at_____°C for_____minutes.

6. Turn off the steam, turn on chilled water to C38, and cool C38 to 20°C.

7. Control the C38 temperature at 20°C for 1 hour.

8. Turn off the C38 agitator and the filter blowdown air.

9. Allow the C38 crystal bed to settle for 1 hour. Control the C38 temperature at 20°C during the settling period.

10. Check that C48 is ready to receive solution.

11. Open the valve from the C38 filter jet to C48.

12. Turn on the jet, and filter the solution from C38 to C48. Control the C48 temperature below 60°C.

13. When the filtration is complete, turn off the filter jet and close the valve.

14. Turn on the C48 agitator.

15. Turn off the C38 chilled water.

16. Immediately add 25 gallons of water to C38 (unless otherwise instructed by supervision) and turn on the chilled water to C38.

17. Turn on the C38 agitator and turn on the filter blowdown air.

18. Check with supervision for handling of the C48 solution.

19. The solution from C48 will normally be jetted back to C17 and combined with the next mainstream feed batch. When this operation is done, proceed as follows:

    a. Check that C17 is ready to receive solution.

    b. Determine the ^{137}Cs content in C17 (either by gamma check sample or from the previous Batch Data Sheet).

    c. Open the valve from C48 to C17 and turn on the jet.

    d. When the C48 liquid level reaches 15%, turn off the C48 agitator.

    e. When C48 is empty, turn off the jet and close the valve.

    f. Check that all valves and jets to and from C48 are closed.

    g. Determine the ^{137}Cs content in C17 by making a gamma check.

    h. By difference (Step g minus Step b), determine the amount of ^{137}Cs recycled from C48 and record it on the Batch Data Sheet.

NOTE: Supervision will determine the number of purification crystallizations to be made.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 TETRAOXALATE CRYSTALLIZATION

NOTE: This procedure is to be done only on approval of supervision. It must be scheduled so that there is a minimum delay between the completion of the _____Cs8 procedure and startup of the _____Cs14 procedure.

A. Preoperational Checks

1. Check that the "Cesium-137 Purification" procedure in C-38 is complete and that C-38 contains at least 20 gallons of solution.

2. Check that C-48 is empty and that all valves and services to C-48 are closed.

3. Check that all valves and services to C-38 are closed except for chilled water and agitation.

4. Check with supervision for approval to start the _____Cs8 operation.

B. Operation

NOTE: Steps B.1--B.3 are done by the operators when the ^{137}Cs content of C-38 is to be determined by sampling. If the ^{137}Cs content of C-38 is to be calculated from material balance, supervision will calculate and the operation will start at Step B.4.

1. Turn off chilled water to C-38; turn on steam to C-38 and heat to 90°C.

2. Control C-38 temperature at 90°C for 1 hour; then pull two 10-milliliter samples and check on the gamma chamber. If the gamma chamber readings do not agree within ±10%, digest C-38 at 90°C for an additional hour and resample. Continue until two samples agree within ±10%; then turn off steam, turn on chilled water, and cool C-38 to 30°C. Control C-38 temperature between 20°C and 40°C.

3. Calculate the ^{137}Cs concentration in C-38 by taking the average of the last two check samples. Determine the volume in C-38 in liters, assuming specific gravity to be 1.05. Calculate the total curies in C-38; then calculate the amount of oxalic acid to be added as follows:

   a. Symbols

      A = curies of ^{137}Cs in C-38,

G = grams of cesium in C-38,
C = cesium concentration in C-38,
V = volume in C-38 (in liters),
W = grams of oxalic acid to be added to C-38.

b. $\frac{\text{A curies}}{\text{33 curies per gram}}$ = G grams.

c. $\frac{\text{G grams}}{\text{V liters}}$ = C* grams per liter.

(*Note: If C is between 11 and 44, proceed; otherwise check with supervision).

d. V liters x (*) x 1.1 = W grams.
(*Check with supervision).

4. Weigh out the oxalic acid and add it (as solid) through the solids-addition funnel to C-38.

5. Rinse the solids-addition funnel with a volume of water equal to 10% of the original volume in C-38 (step 3d).

6. Turn on the steam to C-38 and heat to 70°C.

7. Control C-38 at 70°C ± 2°C for 15 minutes.

8. Turn off the steam to C-38; turn on the chilled water to C-38 and begin cooling. By manual control of the chilled water valves, control the cooling rate so that C-38 cools from 70°C to 30°C in a period of 30 minutes.

9. Continue to cool C-38 and adjust the cooling rate so that C-38 cools from 30°C to 15°C in a period of at least 30 minutes.

10. Digest C-38 at 15°C ± 1°C for 30 minutes.

11. Turn off C-38 agitator and filter blowdown air and allow to settle for 15 minutes. Control C-38 temperature at 15°C ± 1°C.

12. Open the valve from C-38 filter jet to C-48. Turn on the filter jet and filter C-38 to C-48.

13. When C-48 liquid level reaches 10%, turn on C-48 agitator. If C-48 temperature rises above 50°C, turn on the chilled water to C-48 and control the temperature of C-48 between 40°C and 50°C.

14. When C-38 is empty, turn off the filter jet and close the valve from C-38 filter jet to C-48.

15. Immediately pull a 10-milliliter sample from C-48 and check on the gamma chamber. Calculate the total ^{137}Cs content in C-48

(assume specific gravity is 1.1). Inspect the sample for crystals or cloudiness.

a. If the sample is clear and the total ^{137}Cs in C-48 is less than 40% of the original ^{137}Cs content in C-38 (step 3 above), proceed to step 16.

b. If the sample is not clear or if the ^{137}Cs content in C-48 is greater than 40% of the original ^{137}Cs, contact supervision.

16. Add 25 gallons of water to C-38.

17. Turn on the C-38 agitator and agitate C-38 for 1 hour. Control the C-38 temperature between 30°C and 40°C. Do not heat C-38 above 40°C.

18. Pull two 10-milliliter samples from C-38. Inspect the samples for crystals and check them on the gamma chamber. If the samples are clear and the gamma chamber readings agree within ±10%, calculate the total ^{137}Cs in C-38. If samples are not clear or do not agree, digest C-38 an additional hour and recheck.

19. Contact supervision for approval to start _____Cs14 operation.

NOTE: Supervision will direct the recycling of the solution from C-48.

FISSION PRODUCTS DEVELOPMENT LABORATORY

^{137}Cs TETRAOXALATE CRYSTALLIZATION

DATA SHEET

Batch Number _____ Time/Date Started _____

Status (before operations)

Volume C-38 _____ liters

^{137}Cs concentration C-38 (by gamma chamber or _____)

1st check _____ mc/ml 2nd check _____ mc/ml

Average _____ mc/ml

Total curies ^{137}Cs in C-38 _____ Total wt. Cs in C-38 _____ g

Weight concentration Cs in C-38 _____ g/l

Operation

Weight oxalic acid added _____ g

Weight ammonium oxalate added _____ g

Volume rinse water added _____ l

Final volume in C-38 before filtration _____ l

Volume in C-48 after filtration and rinse _____ l

Total curies in C-48 after filtration _____ c

Volume of water added to C-38 after filtration _____ l

Product Data (solution in C-38)

Volume _____ liters

^{137}Cs concentration

1st check _____ mc/ml 2nd check _____ mc/ml

Average _____ mc/ml

Total ^{137}Cs _____ curies

Remarks _____

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 CARBONATE PREPARATION

A. Preoperational Checks

1. Check that P114 and R18 are empty and that all valves and services to P114 and R18 (except R18 off-gas) are closed.

2. Assemble two filters according to the filter buildup procedure.

3. Connect the inlet of the primary filter to the P114 bottom discharge line, using Tygon tubing.

4. Connect the outlet from the primary filter to the inlet of the secondary filter, using Tygon tubing.

5. Connect the outlet from the secondary filter to the nipple on the line from Cell 14E to R18.

6. Check that the manual valve on the line from Cell 14E to R18 is closed and check that the manual bottom discharge valve from P114 is closed.

7. Turn on the vacuum steam jet to R18. If the vacuum gage on R18 reads (-) 20 ft, the jet is operating properly.

NOTE: The operation of turning on the vacuum jet will automatically close the R18 off-gas and turn on the vacuum jet condenser water.

8. Open the manual valve on the line from Cell 14E to R18. If the reading on the R18 vacuum gage remains the same, the filter assemblies are leaktight and the operation can continue. If the vacuum drops, a leak is indicated and the leak must be corrected. Check with supervision.

9. When the filters are leaktight, close the manual valve from Cell 14E to R18 and turn off the R18 vacuum jet (this will automatically open the R18 off-gas and turn off the vacuum jet condenser water).

10. Check the P114 bottom discharge valve by opening it completely; then close it.

11. Make up 5 liters of oxalic acid wash solution as follows: Dissolve 465 grams of oxalic acid in water and dilute to 5 liters.

12. Check that the _____Cs8 operation is complete and that solution is ready to be transferred.

13. Check with supervision for approval to begin operation.

B. Operation

1. Set up communications between Cell 8 panelboard and Cell 14E.

2. Open the valves from C-38 to P114 and transfer _____ liters of solution to P114; then turn off the jet and close the valves. (Check with supervision for the volume to be transferred.)

3. Turn on the P114 agitator and set the speed control at 90%. Agitate for 15 minutes.

4. Pull two 10-milliliter samples from P114 and check on the gamma chamber. If the concentration of ^{137}Cs is between 330 millicuries/milliliter and 1100 millicuries/milliliter, proceed; otherwise contact supervision.

5. NOTE: These samples will be entered in the sample log book as ____Cs14-F1 and _____Cs14-F2, with the ^{137}content by gamma chamber noted. Do not submit the samples to Analytical unless instructed by supervision.

6. Turn off the P114 agitator and determine the volume of solution in P114 (assume specific gravity is 1.1); then turn the agitator back on and set the speed control at 90%.

7. Calculate the total curies of ^{137}Cs in P114.

8. Calculate the amount of oxalic acid to be added as follows:

   a. Symbols

   A = curies of ^{137}Cs in P114,

   G = grams of cesium in P114,

   M_c = moles of cesium in P114,

   M_o = moles of oxalate in P114,

   W_o = equivalent grams of oxalic acid in P114,

   W_f = required grams of oxalic acid in P114,

   V = volume in P114 (in liters),

   W_a = grams of oxalic acid to be added to P114.

b. $\frac{\text{A curies}}{\text{33 curies per gram}} = \text{G grams.}$

c. $\frac{\text{G grams}}{\text{135 grams per moles}} = M_c \text{ moles.}$

d. M_c moles X 2 = M_o moles.

e. M_o moles X 128 grams per mole = W_o grams.

f. V liters X (*) X 1.1 = W_f.
(*Check with supervision)

g. W_f grams minus W_o grams = W_a grams.

Supervision will check calculation.

9. Check with supervision for approval to add the oxalic acid.

10. Weigh out the amount of oxalic acid calculated in step 8.f above and add it (as solid) to P114 through the solids-addition funnel.

11. Rinse the solids-addition funnel with a volume of water equal to 10% of the original volume in P114 (step 6 above).

12. Turn on the steam to P114 and heat to 70°C.

13. Digest P114 at 70°C ± 2°C for 15 minutes.

14. Turn off the steam to P114.

15. Turn on the chilled water to P114 and begin cooling. By manual operation of the chilled water valve, control the cooling rate so that the solution cools from 70°C to 30°C in a period of 30 minutes. Keep the cooling rate as even as possible.

16. Continue cooling P114. Control the cooling rate so that the solution cools from 30°C to 16°C in a period of <u>at least</u> 30 minutes.

17. Digest P114 at 16°C ± 1°C for 15 minutes.

18. Check that all valves to and from R18 are closed.

19. Open the manual valve on the line from Cell 14E to R18.

20. Turn on the R18 vacuum steam jet.

21. Open the bottom discharge valve from P114 and begin filtration. Watch the filters and lines for leaks; if any are noted, close the bottom discharge valve from P114 and contact supervision.

22. Control the temperature of P114 at 16°C ± 1°C during the filtration. When P114 liquid level reaches 10%, turn off the agitator.

23. While filtration is going on, check with supervision to determine if washing of the precipitate is to be done. If washing is to be done, cool the wash solution (step A.12 above) to 15°C, and set up communications between Cell 14E panelboard and P114 solution addition area.

24. Watch the Tygon line between P114 and the primary filter. When no more slurry is being drawn from P114, make five 1-liter washes:

    a. Set P114 agitator speed at 15%.

    b. Add 1 liter of wash solution to P114.

    c. Allow all solution to be pulled from P114.

    d. Repeat steps a, b, and c for a total of five washes.

25. Continue to pull vacuum on R18 for 30 minutes after the last of the solution has been drawn from P114. Then turn off the vacuum steam jet on R18 and close the manual valve between Cell 14E and R18. Leave the P114 bottom discharge valve open.

26. Turn on R18 air-sparger and sparge for 5 minutes; then turn air off.

27. Open the valves on the R18 sample lines and pull two samples. Check the samples on the gamma chamber and calculate the total ^{137}Cs in R18 (assume specific gravity is 1.1). If the ^{137}Cs content in R18 is less than 40% of the original ^{137}Cs content in P114 (step B.7 above), proceed; otherwise, contact supervision for recycle instructions.

28. Check that C-48 is empty except for previous oxalate filtrates and that there is sufficient free volume in C-48 to accept the solution from R18.

29. Open the valves from R18 to C-48, jet R18 to C-48; then turn off the jet and close the valves.

30. Close the bottom discharge valve from P114.

31. Release the clamp from the cover of the primary filter and carefully remove the cover.

32. NOTE: If any white solid adheres to the gaskets, scrape it off into the filter thimble.

33. Using the special filter tongs, lift the filter thimble out of the stainless steel filter shell.

34. Store the precipitate in the thimble in a stainless steel beaker until it is ready to calcine.

C. Calcination

1. Check that the Cell 14-W furnace is cool (<110°C).

2. Check that the nickel support rack is in place in the furnace.

3. Using a spatula, remove the precipitate from the filter thimble and put it into a nickel calcining tray. Set the filter thimble aside for cleanup and recovery of the ^{137}Cs.

4. Transfer the loaded calcining tray to Cell 14W.

5. Insert the calcining tray into the furnace by positioning to furnace loading platform in the furnace door, lifting the calcining tray onto one of the shelves of the platform, and then sliding the tray into the furnace until it just clears the door.

NOTE: More than one batch of precipitate may be calcined at once. Check with supervision.

6. Remove the furnace loading platform. Close and seal the furnace door.

7. Check that the air inlet valve to the furnace is open and that the N_2 inlet to the furnace is closed.

8. Connect the off-gas outlet from the furnace to the off-gas nipple (not to the scrubber) and open the manual valve on the off-gas header.

9. Check that the furnace controller is set at the same temperature as the furnace.

10. Turn on the furnace power.

11. Manually set the furnace controller at 20°C above furnace temperature. As the furnace begins to heat, advance the controller every 5 minutes and keep it at 20°C above furnace temperature.

12. When the furnace temperature reaches 110°C, control at 110°C for 2 hours.

13. After 2 hours at 110°C, manually increase the set point on the furnace controller as in step C.12 above and heat the furnace to 300°C.

14. Control the furnace temperature at 300°C for 2 hours.

15. Turn off the furnace power switch and allow the furnace to cool.

16. When the furnace has cooled to less than 170°C, open the furnace door and close the manual valve on the off-gas header.

17. When the furnace has cooled to less than 120°C, remove the calcining trays. Transfer the trays to Cell 14E..

18. Stir the calcined powder to break up any lumps. Transfer the powder from the calcining tray into a tared stainless steel storage beaker and determine the total weight of powder.

19. Weigh out a sample of the powder into a calorimeter cup and run calorimetric analysis.

20. Check with supervision for storage of powder.

FISSION PRODUCTS DEVELOPMENT LABORATORY

^{137}Cs CARBONATE PREPARATION

DATA SHEET

Batch Number_____ Time/Date Started_____

Status (before operations)

Volume in P114_____liters

^{137}Cs concentration in P114 (by gamma or_____)

1st check_____mc/ml 2nd check_____mc/ml

Average_____mc/ml

Total curies ^{137}Cs in P114_____ Total wt. Cs in P114_____g

Operation

Weight oxalic acid added_____g

Digestion Times: Started_____ Completed_____

Cooling Times: Started_____ Completed_____

Filtration Times: Started_____ Completed_____

Filtrate Volume_____liters

^{137}Cs concentration in filtrate

1st check_____mc/ml 2nd check_____mc/ml

Average_____mc/ml

Calcination Times: Started_____ Completed_____

Calcination temp._____(attach furnace record sheets)

Product Data

Total weight_____

Calorimeter Results

1st check: Sample wt_____g, cal. temp._____ m.v., c/g_____

2nd check: Sample wt_____g, cal. temp._____ m.v., c/g_____

Average_____c/g

Total curies ^{137}Cs in product_____

Product disposition_____

Remarks_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

FURNACE RECORD

Date _____

Material _____ Batch No. _____

Furnace No. _____ Operation _____

Remarks _____

Date	Time	Controller Set Point	Controller Temperature	Recorder Temperature	Cell Temp.	Initials and Remarks

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 CHLORIDE PREPARATION

A. Preliminary Setup

1. Check that the following items are in Cell 13:

   a. Three 800-milliliter tantalum beakers.

   b. Tantalum stirring rods.

   c. Hot plates with asbestos pad.

   d. Balance and weights.

   e. Two settling vessels, A and B.

   f. Vacuum flask with lines.

   g. Off-gas bell jar.

   h. Glass stick filters (at least 6) with ball joints.

   i. Filter storage beaker.

   j. 50-milliliter graduated cylinder.

2. Check that the following items are in Cell 14E:

   a. Hot plate with off-gas funnel connected to off-gas line with manual valve closed.

   b. Variac-control hot plate.

   c. Slanted beaker holder.

   d. Tantalum stirring rod.

   e. Storage cans as required (check with supervision).

   f. Balance, weights, and spatulas for weighing powder.

3. Assemble (or check) the Cell 13 off-gas scrubber system as follows:

NOTE: The NaOH solution in the scrubbers is changed every second batch.

   a. Drain solution from both NaOH scrubbers and discard.

   b. Empty condensate collection flask. Neutralize by adding an equal volume of 10 M NaOH; then discard. Replace flask.

c. Fill both NaOH scrubbers to the overflow arm with 4 M NaOH.

d. Connect water to bottom nipple of condenser coil. Check that the line from the top nipple is directed to drain.

e. Connect condenser off-gas to the gas inlet of the first NaOH scrubber.

f. Connect the gas outlet from the first NaOH scrubber to the gas inlet of the second NaOH scrubber.

g. Connect the gas outlet from the second NaOH scrubber to the in-cell vacuum pump.

h. Connect the vacuum pump outlet to off-gas.

4. Insert a 500-milliliter "squeeze" bottle of concentrated HCl.

5. Check with supervision for identification of the $^{137}Cs_2CO_3$ powder to be used.

B. Dissolving of $^{137}Cs_2CO_3$

1. Weigh out_____grams of $^{137}Cs_2CO_3$ and put into a tantalum beaker.

2. If there is a heel in settling vessel A left from a previous run, drain the heel into the tantalum beaker containing the $^{137}Cs_2CO_3$ (no rinse of vessel A required).

3. Add demineralized water to the beaker until it is approximately half full. Stir continuously for 10 minutes.

4. Check that the bottom outlet from vessel A is closed; then slurry the contents of the tantalum beaker into vessel A (no rinse of beaker is required). Set the tantalum beaker aside for the next batch.

5. Allow the slurry in vessel A to settle for 30 minutes. While the slurry is settling, check that the vacuum transfer flask is empty; then connect the outlet from the vacuum transfer flask to R18. Connect a glass filter stick to the vacuum transfer flask inlet.

6. After the 30-minute settling, open the manual block valve (in Cell 14E) to R18 and turn on the R18 vacuum jet.

7. Filter the solution from the top portion of settling vessel A into the vacuum transfer flask as follows:

   a. Hold the filter stick just under the surface of the solution. As the solution level drops, lower the filter.

b. Keep the filter at the side of the vessel; do not let the filter get into the lower (narrow) portion of the vessel.

c. If the filter becomes plugged, lift it out of the solution but hold it over the vessel. Turn off vacuum and allow any solution that is in the filter to drain back into the settling vessel. Then disconnect the filter and store it in the filter storage beaker. Attach a new filter and proceed.

8. When the solution from the top portion of settling vessel A has been filtered, turn off vacuum (let any solution from filter drain back to the settling vessel) and disconnect the filter. Store the filter in the filter storage beaker. Close the R18 block valve.

9. Transfer the solution from the vacuum transfer flask to a clean tantalum beaker.

NOTE: If there is enough solution to more than half fill the tantalum beaker, divide the solution into two approximately equal portions and put each portion in a tantalum beaker.

C. Chloride Conversion

NOTE: If two beakers are used, treat both by same procedure.

1. Measure approximately 25 milliliters (±5 milliliters, since volume is not critical) of concentrated HCl into the 50-milliliter graduated beaker.

2. While stirring the filtered Cs_2CO_3 solution in the tantalum beaker (use tantalum stirring rod), slowly add the ~25 milliliter of concentrated HCl. There will be some foaming. After adding the HCl, stir until foaming stops.

3. Repeat steps 1 and 2 until there is no foaming upon addition of ~25 milliliters of HCl. Then stir well and allow the solution to stand for 10 minutes.

4. Without stirring, add one more ~25-milliliter portion of concentrated HCl and observe carefully for foaming. If no foaming occurs, stir slowly and observe. If no foaming occurs, proceed to step 5. If foaming occurs at any time, stir the solution well and allow to stand for 10 minutes. Repeat this step until no foaming occurs.

5. Stir the solution and slurry the contents of the tantalum beaker into settling vessel B (no rinse of beaker is required). Set the tantalum beaker aside for the next batch.

NOTE: Settling vessel B may contain a heel from a previous batch - leave heel in vessel B.

6. Allow the slurry in vessel B to settle for 30 minutes. While the slurry is settling, check that the vacuum transfer flask is empty; then connect the outlet from the vacuum transfer flask to R18. Connect a clean glass filter stick to the vacuum transfer flask inlet.

7. After the 30-minute settling, open the manual block valve (in Cell 14E) to R18 and turn on the R18 vacuum jet.

8. Filter the solution from the top portion of settling vessel B into the vacuum transfer flask as follows:

   a. Hold the filter stick just under the surface of the solution. As the solution level drops, lower the filter.

   b. Keep the filter at the side of the vessel; do not let the filter get into the lower (narrow) portion of the vessel.

   c. If the filter becomes plugged, lift it out of the solution but hold it over the vessel. Turn off vacuum and allow any solution to drain back into the settling vessel. Then disconnect the filter and store it in the filter storage beaker. Attach a new filter and proceed.

9. When the solution from the top portion of settling vessel B has been filtered, turn off R18 vacuum (let any solution from filter drain back into the settling vessel) and disconnect the filter. Store the filter in the filter storage beaker. Close the R18 block valve.

10. Transfer the solution from the vacuum transfer flask to a clean tantalum beaker.

D. Evaporation

1. Set the tantalum beaker containing the filtered $^{137}CsCl$ solution on the hot plate.

2. Set the off-gas bell jar over the tantalum beaker, allowing the bottom edge of the bell jar to rest on the asbestos pad.

3. Connect the gas outlet from the bell jar to the gas inlet of the off-gas condenser

4. Turn on the cooling water to the off-gas condenser and check that the water is flowing (just a steady stream -- no measured flow is required).

5. Turn on the in-cell vacuum pump.

6. Turn on the hot plate and set on "medium."

7. While the solution is heating, observe the system for evidences of leaks, especially vapor coming out of the joint between the off-gas condenser and the condensate flask. If any vapor is noted, reset the joints of the off-gas system. If leaks cannot be stopped, turn off the hot plate and contact supervision.

8. Allow the solution in the tantalum beaker to evaporate to dryness. Check occasionally for evidence of leaks in the off-gas system. After 5 hours, check the beaker by lifting the bell jar. If the powder is not completely dry, replace the bell jar and continue evaporation, with checks every hour, until the powder is dry.

9. When the powder is dry, turn off the hot plate. Disconnect the off-gas outlet of the bell jar from the off-gas system and remove the bell jar from over the beaker.

10. Turn off the vacuum pump and turn off the water to the off-gas condenser.

E. Re-evaporation and Baking

1. Transfer the tantalum beaker containing the dried $^{137}CsCl$ to Cell 14E.

2. Add demineralized water to the beaker until the beaker is approximately one-half full. Stir to dissolve the powder.

3. Set the beaker on the hot plate that has the off-gas funnel. Position the funnel so that it covers the top of the beaker, and open the off-gas valve.

4. Turn on the hot plate and set on "medium."

5. Check the solution every 15 minutes. When the solution becomes syrupy, remove the beaker from the hot plate and set it in the slanted beaker holder. Turn off the hot plate and close the off-gas valve.

6. Immediately begin stirring the powder, using the tantalum stirring rod. Stir continuously until the powder is no longer sticky (usually about 15 minutes). Allow the beaker to stand for an additional 45 minutes, stirring for 5 minutes out of every 15 minutes.

7. Transfer the beaker to the other hot plate. Turn on the hot plate and set control at 50°C.

8. Begin stirring powder once every 30 minutes (stir for 5 minutes each time); check powder temperature at each stirring, using the stainless steel thermocouple.

   a. Heat the powder to 350°C ± 50°C.

b. At each stirring, check for formation of lumps and break up any lumps that form.

c. Continue heating and stirring for a total of 4 hours after the powder temperature reaches 350°C.

9. After the powder has been baked, turn off the hot plate.

10. Weigh the tantalum beaker and powder. Transfer the powder from the tantalum beaker to the specified storage can, and close the can. Re-weigh the tantalum beaker and determine powder weight by difference.

11. Transfer the tantalum beaker and stirring rod back to Cell 13.

F. Cleanup Operations

NOTE: The operations in this section are to be done after completion of a series of batches or as otherwise instructed by supervision.

1. Drain the slurry heel from settling vessel A into the tantalum beaker that was used for the $^{137}Cs_2CO_3$ dissolving (Section B above).

2. Set the beaker from step 1 under settling vessel B. Carefully drain settling vessel B into the beaker -- there will be some foaming. Set the beaker aside temporarily.

3. Add approximately 200 milliliters of demineralized water to the tantalum beaker that was used for the chloride conversion (Section C above).

4. Check that the bottom drain on settling vessel A is closed. Swirl the water in the tantalum beaker (from step 3 above) to wash the sides, then pour the water into vessel A. Set the beaker under vessel A and drain the solution from vessel A into the beaker.

5. Repeat step 4 using settling vessel B (use the same water).

6. Rinse all used filter sticks and tantalum stirring rods by swirling them in the water in the tantalum beaker (from step 5).

7. Pour the contents of the tantalum beaker from step 6 into the tantalum beaker containing the heels from the settling vessels (see step 2 above).

8. Refer to Section C above. Perform steps C.1 through C.4 on the solution in the tantalum beaker from step 7.

9. Refer to Section D above. Perform all of Section D on the solution from step 8.

10. Identify the dried powder as "^{137}CsCl Residue." Check with supervision for storage instructions. (Several batches of residue will generally be accumulated before being reprocessed).

FISSION PRODUCTS DEVELOPMENT LABORATORY

$^{137}CsCl$ PREPARATION

DATA SHEET

Batch Number_____ Time/date started_____

Status (before operations)

$^{137}Cs_2CO_3$ Batch Number_____

$^{137}Cs_2CO_3$ Activity concentration_____ c/g

Operation

Weight $^{137}Cs_2CO_3$_____ g

$^{137}Cs_2CO_3$ Dissolving

Time filtration started_____

Time filtration completed_____

Total filtration time_____ min

Chloride Conversion

Total volume HCl added_____ ml

Time filtration started_____

Time filtration completed_____

Total filtration time_____

Re-evaporation and Baking

Time evaporation started_____

Time baking (350°C) started_____

Time baking completed_____

Product Disposition

Sample code_____

Storage_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CESIUM-137 CHLORIDE PELLET PRESSING

A. Preliminary Setup

1. Check that the following items are in the cell; insert new items as necessary:

   a. Pellet press frame with ram.

   b. Die support shim if required.

   c. Hydraulic pump, gage (10,000 psi), and hoses.

   d. Die with punches. Check with supervision for die size.

   e. Die saddle or manipulator band.

   f. Ejection stand.

   g. Three ejection punches (two that are 1 1/2 inches longer than the die and one that is 1/4 inch longer than the die).

   h. Stearic acid solution (0.1 gram of stearic acid in 100 milliliters of acetone).

   i. Q tips.

   j. 1-liter stainless steel beaker with grips.

   k. Stainless steel weighing beaker.

   l. Stainless steel spoons.

   m. Stainless steel funnel (sized to the die) with rod.

   n. Balance (check with supervision).

   o. Stainless steel mortar and pestle.

   p. Hot plate.

   q. Storage tray for pellets if required.

   r. Measuring equipment or go-no-go gages as required.

2. Prepare powder as follows:

   a. Check with supervision for designation of the $^{137}CsCl$ batch to be used and for amount of powder required.

b. Transfer the designated powder from storage to the 1-liter stainless steel beaker.

c. Check the powder for lumps. If necessary, use the mortar and pestle to break up lumps; then return powder to the beaker.

d. Connect the electrical lead on the hot plate and position the hot plate at the side of the cell so that it does not interfere with other operations. Label the hot plate switch at the cell face.

e. Turn the hot plate on "low". Set the beaker containing the $^{137}CsCl$ on the hot plate (note time that $^{137}CsCl$ is placed on hot plate).

3. Arrange press assembly as follows:

a. Position the press frame in the center of the cell. Level frame with shims.

b. Position the hydraulic pump so that the valves are operable with manipulators and the gage is visible. Connect hydraulic hose to the ram.

c. Center the die support shim on the press base if required.

d. Connect the electrical lead to the pump.

e. Set a dummy die on the press; turn on the pump and check for steadiness; then apply 500 psig pressure. Check for hydraulic leaks and relieve pressure.

f. Turn off the pump and remove the dummy die.

g. Label the pump switch at cell face.

4. Arrange balance, weights, weighing beaker, spoons, funnel, and die parts for easy access.

5. Check with supervision for pellet-handling instructions.

a. If pellets are to be stored on trays, arrange trays to receive pellets.

b. If pellets are to be loaded directly into capsules or shipping cans, insert the proper number of containers into the cell. (Supervision will arrange for degreasing, marking, etc., prior to start of pellet pressing.)

B. Pellet Pressing

1. Check with supervision for approval to begin operation. The $^{137}CsCl$ must be dried for 4 hours before pellet pressing is started. Supervision will write up details concerning number of pellets, weights, etc. Be sure that all "Special Instructions" information is available before starting operation.

2. Lubricate the die bore and the long punch with stearic acid, using a Q tip.

3. Insert the short punch into the die and make sure that the punch is flush with the bottom of the die.

4. Set the die into the die saddle.

5. Place the funnel into the die.

NOTE: Steps 6, 7, and 8 must be done in one continuous operation. If it becomes necessary to stop operation for more than 10 minutes after a die load has been weighed out (but before it is put into the die), return the powder to the beaker on the hot plate.

6. Weight out_____grams of powder from the beaker on the hot plate into the weighing beaker.

7. Transfer the weighed powder from the weighing beaker through the funnel into the die. Check that all powder goes into the die; use the funnel rod if necessary to clear the funnel. Remove funnel.

8. Insert the long punch into the die. Press down the punch with the manipulator fingers to seat it.

9. Set the assembled die and saddle on the press base and center it.

10. Turn on the hydraulic pump and begin lowering the ram. When the ram makes contact with the top punch, stop and check the alignment of the system. Reset if necessary.

11. Slowly increase the pressure. As long as there is visible movement of the top punch, maintain just enough pressure to continue movement. When visible movement has stopped, increase the pressure to_____psig and hold for_____minutes.

12. Release the pressure by opening the pump valve. Turn off the pump.

13. Remove the die and saddle from the press.

14. Position the ejection stand in the center of press base.

15. Remove the die from the saddle. Set the die on the ejection stand, top punch up.

16. Turn on the pump. Lower the ram until it contacts the top punch. Gradually increase the pressure until the punch begins to move; then maintain just enough pressure to continue movement until the pellet is ejected.

NOTE: If the pressure reaches a value equal to one-half the pressing pressure (Step 11 above) before the punch moves, hold and contact supervision.

17. After the pellet has been ejected, release the pressure and turn off the pump.

18. Remove the die from the ejection stand.

19. Remove the pellet from the ejection stand. Weigh, measure, and load or store the pellet as instructed by supervision.

20. Invert the die body in the ejection stand so that the long punch is down.

21. Hold the short ejection punch centered in the die. Turn on the pump and apply pressure until the ejection punch is about two-thirds into the die body. Then release pressure, replace the short ejection punch with the long ejection punch, and apply pressure until there is a sudden release of pressure, indicating that the punch is out of the die. Release pressure immediately.

22. Remove the die and punches from the ejection stand, and remove the ejection stand from the press.

FISSION PRODUCTS DEVELOPMENT LABORATORY

PELLET FABRICATION DATA

Material _____ Date _____

Powder Batch _____ Powder Specific Activity _____ c/g

(NOTE: Use Separate Sheet for Different Powder Batches)

Die Size _____ Weight per Pellet _____ g

First Pressure _____ Second Pressure _____

Remarks: _____

Pellet No.	Pressed		Weight Powder	First Pressure	Second Pressure	Initials and Remarks
	Date	Time				

FISSION PRODUCTS DEVELOPMENT LABORATORY

FINISHED PELLET DATA

Material _____ Date _____

Powder Batch _____ (Use separate sheets for pellets from different powder)

Die Size _____ Max. Sintering Temp. _____ Sintering Time _____

Remarks _____

Pellet No.	Weight	Height	Diameter	Heat* Output	Initials and Remarks

*Attach Calorimetric Record Sheets

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

UNLOADING OF STRONTIUM-90 FROM HAPO CASKS

A. Preoperational Checks

1. Enter Cell 15 and check that all the following manual valves in the unloading station are closed:

   a. To W19 and W20.

   b. Off-gas.

   c. Vacuum to STT.

   d. From R11.

   e. Cell 15 to E16-P45-P12.

2. Close all valves on Cell 1 panelboard.

3. Check with supervision for approval to open Cell 1-N.

4. Remove the blocks from Cell 1-N. Enter Cell 1-N under cell entry procedures and check that all the following manual valves in the unloading station are closed:

   a. STT to off gas.

   b. STT to J56-1 and J64-1.

   c. Feed to STT.

   d. Feed to HAPO.

   e. HAPO to J56-1 and J64-1.

5. Open Cell 1-N cell ventilation damper.

6. Check that the following items are clean and in good condition:

   a. Movable shields (armor plate and plastic) for Cell 1-N.

   b. Special tool for removing the outer cap to the HAPO vent valve.

   c. Vacuum lifting tool for vent valve inner cap.

   d. Snaptite handling tool with adapter for HAPO Snaptite blank.

   e. Long wrench (5/8 inch hexagonal) for process line cap bolts and impact wrench with 5/8-inch hexagonal adapter.

f. Lifting tool for process line caps.

g. HAPO-connector lifting tool.

h. HAPO-connector operating tool.

i. Long Allen wrench (1/2 inch) for HAPO connectors.

j. Special hook for opening vent valve.

k. Standard long tongs and hooks (2 each).

l. Ladders for cell entry.

m. Contamination zone supplies and cell entry supplies.

n. HAPO connector gaskets (12), and valve cover gaskets (6).

7. Check that P23 and P34 are empty.

8. Rinse M131 as instructed by supervision.

9. Make up 50 gallons of_____N HNO_3 in M131. Check with supervision for makeup.

10. Check T.V. camera tilt, pan, and focus.

B. Cask Handling

1. When the cask is received at FPDL, Health Physics will make survey and smear the cask. Record the data on Data Sheet No. 1. Wrap the cask in plastic, leaving the cupola and cooling coil nozzles uncovered.

2. Just before inserting the cask into Cell 1-N, open the cooling line outlet in the cell and blow the condensate from the lines.

3. Insert the cask into Cell 1-N and position it as instructed by supervision.

4. Insert the ladders for personnel entry into Cell 1-N. Equipment for personnel entry is as follows:

a. Coveralls.

b. Rubber gloves.

c. Shoe covers.

d. Assault mask.

e. Pencil meters, dosimeters, screamer.

f. Safety line.

5. Enter Cell 1-N and attach the coil pressure-test device.

6. Remove the thermowell cap and attach the thermocouple to the cask.

7. Remove the blank flanges from the cooling coil nozzles, and connect the inlet and outlet cooling lines to the cask. Check that the manual valve on inlet to cask coil is open and the bypass valve is closed.

8. Evacuate Cell 1-N.

9. Check the temperature of the filter and contact supervision.

NOTE: Supervision will determine the cooling procedure to be used.

C. Cask Connection

1. Check with supervision that the cooling process is complete and cell entry is authorized.

2. Install the armor plate shield and safety rails.

3. Enter Cell 1 under cell entry procedure (plastic suit) and remove the nuts from the cupola cover. Attach the choker and leave the cupola cover in place.

4. Remove the cover from the vent valve well. Set the cover and the nuts from the cupola cover on the cask (clear of the cupola cover). Survey the cover and well.

5. Check that the HAPO connectors are in place on the HAPO connector rack.

6. Check that the lifting screws are in place on the vent valve outer cap.

7. Evacuate Cell 1.

8. Install the plastic shield.

9. Provide the following protective equipment:

a. Face shields, gloves, shoe covers, and safety glasses.

b. Pencil meters, two dosimeters, and screamer.

10. Set up work area around Cell 1 as follows:

a. Position the beta monitron over the opening.

b. Set up contamination zone to receive tools.

NOTE: Radiation Work Permit regulations will apply.

11. Remove the vent valve outer cap by loosening the large hold-down bolt and then attaching the special lifting tool and rotating the cap.

12. Lift the vent valve outer cap from the cell. Survey the cap and wrap it in plastic.

13. Lift the vent valve inner cap from the cell using the vacuum lifting device or the special snare. Survey the cap and wrap it in plastic.

14. Disconnect the blank female Snaptite from the vent line and remove it from the cell. Survey the Snaptite and wrap it in plastic.

15. Insert the handling tool in the Snaptite adapter, and connect the vent line to hot off-gas.

16. Open the cask vent valve, using the special tool.

17. Open the valve (on the panelboard) on the vent line and record the pressure on the cask on Data Sheet No. 1.

18. Open the manual valve on the vent line and vent the cask to hot off-gas. Use a long tool for this operation.

19. Remove the plastic shield. Lift the cupola cover out of the cell and replace the plastic shield. Survey the cupola cover and wrap it in plastic.

20. Remove the caps from the "solution in" and "slurry in" process nozzles by the following procedure:

    a. Check supervision for designation of the nozzles.

    b. Remove the bolts from the outer cap (using the long manual wrench or impact wrench).

    c. Connect the lifting tool to the outer cap, rotate the cap, and lift it out of the cell.

    d. Survey the bolts and cap and wrap them in plastic.

    e. Remove the gasket, using long tongs.

21. Remove the blank HAPO connector from the "solution in" process nozzles by the following procedures:

    a. Loosen the connector, using the Allen wrench.

    b. Attach the lifting tool to the connector and lift it out of the cell. Survey the connector and wrap it in plastic.

c. Remove the gasket with tongs.

d. Insert a new gasket onto the nozzle.

22. Attach the handling tool to the HAPO connector on the line from R11.

23. Set the connector (R11 line) on the "solution in" nozzle. Insert the Allen wrench and tighten the HAPO connector. Remove the handling tool.

24. Remove the blank HAPO connector from the "slurry in" process nozzle as follows:

    a. Loosen the connector, using the Allen wrench.

    b. Attach the lifting tool to the connector and lift it out of the cell. Survey the cap and wrap it in plastic.

    c. Remove the gasket with tongs.

    d. Insert a new gasket onto the nozzle.

25. Attach the handling tool to the HAPO connector on the line to P34 (P23).

26. Set the connector (P34, P23 line) on the "slurry in" nozzle. Insert the Allen wrench and tighten the HAPO connector. Remove the handling tool.

27. Close the cask vent valve and the valve to hot off-gas (on the panelboard).

28. Insert a hose into Cell 1-N and fill the cupola of the cask with <u>demineralized water</u>. Fill to a point above the connectors.

29. Close all openings in the plastic shield. Isolate the work area over Cell 1-N.

    No entry will be allowed while the connector test is made.

30. Adjust the T.V. camera to get a view of the water level in the cupola.

31. Check that all valves on the Cell 1 panelboard are closed.

32. Open the valve from the P34 (P23) jet to_____ only (check with supervision).

33. Turn on the P34 (P23) jet, and use the T.V. camera to observe the water level in the cupola.

34. Operate the jet for 3 minutes. If the water level stays the same, proceed to step 36 below.

35. If water level drops during step 34, a leak in one of the connections is indicated. Continue to jet until the water level stops dropping; then turn off the jet and close the valve. Then proceed as follows:

    a. Open the valve on the line from Area 31 to line 1016 (on the panelboard) and open the manual valve on "Feed to HAPO" line.

    b. Survey top of Cell 1 before entry.

    c. Begin adding demineralized water from Area 31 to line 1016 at 2 gallons per minute and observe the water levels in the cupola and in the in-cell funnel to line 1016.

    d. Continue adding water until the in-cell funnel is almost full; then stop addition and observe the water levels. If water flows from the funnel and accumulates in the cupola, a leak in the R11 to HAPO line is indicated. If the water level in the funnel remains constant, the leak is in the HAPO to P34 (P23) connection.

    e. Close the valve on line 1016.

    f. When the leakage point has been identified, check with supervision for approval to enter the work area over Cell 1-N. A new radiation survey must be made on entry. Disconnect the leaking connector and reconnect as follows:

        (1) Attach the handling tool to the HAPO connector.

        (2) Loosen the connector with the Allen wrench and lift it off the process nozzle.

        (3) Remove the gasket with tongs, then insert a new gasket.

        (4) Set the connector back on the nozzle and tighten it with the Allen wrench.

        (5) Remove the handling tool.

        (6) Repeat the leak test, starting at step 28 above.

        NOTE: If a leak is detected in the R11 to HAPO connection, this must be corrected before it can be determined whether the HAPO to P34 (P23) connection is also leaking. When the system has been made leaktight, Health Physics will resurvey area over Cell 1-N.

36. After Health Physics has surveyed the area over the cell, open the manual valve on the feed to HAPO line (line from R11) and remove all tools from the cell. Remove the armor plate and plastic shields. Replace and seal the shielding blocks.

37. Adjust Cell 1-N cell ventilation to -1.5 inches of H_2O.

D. Slurry Operation

1. Add 50 gallons of demineralized water to R11.

2. Open the valve from the P34 (P23) jet to_____ only (check with supervision).

3. Open the following valves from R11 to the cask:

   a. R11 to pump.

   b. Pump by-pass.

   c. Pump discharge.

4. Turn on the P34 (P23) jet.

5. Record the following every 5 minutes on Data Sheet No. 2:

   a. R11 volume.

   b. R11 pressure.

   c. P_____volume.

   d. P_____pressure.

   e. Cell 1 electrometer reading.

   f. Filter temperature.

6. When P34 (P23) volume reaches 10%, turn on agitator and cooling water.

7. When R11 volume reaches 10%, begin the addition of 50 gallons of _____N HNO_3 from M131 to R11 at a rate of 3 gallons per minute.

8. When R11 is empty, turn off the P34 (P23) jet. Close the valve from the jet to P34 (P23).

9. Close the following valves from R11 to the cask:

   a. R11 to pump.

   b. Pump by-pass.

   c. Pump discharge.

E. Sampling of Strontium Solution

1. Agitate P34 (P23) for 30 minutes after slurry operation is complete.

2. Turn off the chilled water to P34 (P23). Turn on the steam and heat P34 (P23) to_____°C for_____minutes; then turn off the steam (check with supervision for temperature and time).

3. Cool P34 (P23) to_____°C and take three samples.

   Codes:_____Sr_____-S1-1,_____Sr_____-S1-2 and, _____Sr-

   _____-S1-3.

4. Rush sample to Analytical and request analysis on ^{90}Sr and ^{89}Sr.

5. Turn on the chilled water and agitator to P34 (P23).

6. Check with supervision regarding storage of solution and tagging of tanks.

F. Rinse of Cask

1. Make up 50 gallons of 2 N HNO_3 in M131 as follows:

   Dilute 25 liters of 70% HNO_3 to 50 gallons with demineralized water.

2. Add the 50 gallons of 2 N HNO_3 from M131 to R11.

3. Open the valve from the P34 (P23) jet to_____ only (check with supervision).

4. Open the following valves from R11 to the cask:

   a. R11 to pump.

   b. Pump by-pass.

   c. Pump discharge.

5. Turn on the P34 (P23) jet.

6. Record the following every 5 minutes on Data Sheet No. 2:

   a. R11 volume.

   b. R11 pressure.

   c. P_____volume.

   d. P_____pressure.

   e. Cell 1 electrometer reading.

   f. Filter temperature.

7. When P34 (P23) volume reaches 10%, turn on the agitator and cooling water.

8. While the acid is being jetted, add 50 gallons of demineralized water to M131.

9. When R11 volume reaches 5%, start adding 50 gallons of demineralized water from M131 to R11 at 3 gallons per minute.

10. When R11 is empty, do the following:

    a. Turn off the P34 (P23) jet.

    b. Close the valve from the jet to_____.

    c. Leave the valves from R11 to the cask OPEN.

11. Take two samples from_____.

    Codes:_____Sr_____-S2-1, _____Sr_____-S2-2.

12. Rush samples to Analytical and request analysis on ^{90}Sr.

NOTE: Supervision will determine the disposition of the rinse solution and direct any further rinsing if necessary.

G. Backwash of Filter

1. When it has been determined that the cask is empty and the rinses are complete, remove the shielding blocks from Cell 1 under Radiation Work Permit regulations and insert the armor plate and plastic shields.

2. Open the cask vent valve and the valve to hot off-gas (on the panelboard).

3. Remove the outer cap from the "filtrate out" process nozzle by the following steps:

    a. Remove the bolts from the outer cap (using the long manual wrench or the impact wrench).

    b. Connect the lifting tool to the outer cap and lift it out of the cell.

    c. Survey the bolts and cap and wrap them in plastic.

    d. Remove the gasket, using long tongs.

4. Remove the blank HAPO connector from the "filtrate out" nozzle as follows:

a. Loosen the connector, using the Allen wrench.

b. Attach the lifting tool to the connector and lift it out of the cell. Survey the connector and wrap it in plastic.

c. Remove the gasket, using tongs.

d. Insert a new gasket onto the nozzle.

5. Attach the handling tool to the HAPO connector on the line from R11 (this line is now connected to the "solution in" nozzle).

6. Loosen the connector, remove it from the "solution in" nozzle, and set it onto the "filtrate out" nozzle. Tighten the connector, and then remove the handling tool.

7. Insert a new gasket onto the "solution in" nozzle.

8. Attach the handling tool to a blank HAPO connector.

9. Set the blank HAPO connector onto the "solution in" nozzle and tighten. Remove the handling tool.

10. Close the cask vent valve and the HAPO to off-gas valve on the panelboard.

11. Remove all tools from the cell, and seal the plastic shield. Isolate the work area over Cell 1-N; no entry will be allowed while the backwash is being done.

12. Add 50 gallons of water to R11.

13. Open the following valves from R11 to the cask:

    a. R11 to pump.

    b. Pump discharge.

    c. Pump by-pass.

14. Open the valve from the P34 (P23) jet to_____ only (check with supervision) and open the valve from the cask to the P34 (P23) jet.

15. Turn on the P34 (P23) jet and transfer the 50 gallons of water from R11 through the cask to_____.

16. When R11 is empty, turn off the jet and close the valves from cask to jet and from jet to_____. Leave the valves from R11 to the cask open.

17. Check with supervision for approval to enter the work area over Cell 1-N. A new radiation survey must be made before entry can be permitted.

18. Unseal the plastic shield.

19. Open the cask vent valve and the valve on the cask to off-gas line on the panelboard.

20. Attach the handling tool to the connector on the line to the P34 (P23) jet (this line is now on the "slurry in" nozzle). Loosen the connector, remove it from the nozzle, and set the connector into the storage rack; then remove the handling tool.

21. Remove the used gasket from the "slurry in" nozzle and insert a new gasket.

22. Attach the handling tool to the connector on the line from R11 (this line is now on the "filtrate out" nozzle), loosen the connector, and remove it from the nozzle.

23. Set the connector on the line from R11 onto the "slurry in" line. Tighten the connector with the Allen wrench, and then remove the handling tool.

24. Remove the used gasket from the "filtrate out" nozzle and insert a new gasket.

25. Attach the handling tool to the connector on the line to the P34 (P23) jet (this line is now in the storage rack).

26. Set the connector on the line to the P34 (P23) jet onto the "filtrate out" nozzle. Tighten the connector with the Allen wrench and remove the handling tool.

27. Close the cask vent valve and the valve from cask to off-gas on the panelboard.

28. Remove tools from the cell and seal the plastic shield.

NOTE: The heating and cooling cycles are different for the two types of casks (HAPO-I and HAPO-II); therefore the cask drying procedures and coil tests differ. Permission must be obtained from supervision to start the drying procedure.

H. HAPO-I Cask Drying Procedure and Coil Leak Testing

1. Close the following valves from R11 to the cask:

   a. R11 to pump.

   b. Pump by-pass.

   c. Pump discharge.

2. Open the valve on the solutions addition to line 1016.

3. Turn off the chilled water to the cask coil.

4. Open the valve from the cask to the P34 (P23) jet and the valve from the P34 (P23) jet to_____ only.

5. Turn on the P34 (P23) jet.

6. Turn on steam to the cask coil and heat the cask to 110°C. Leave the P34 (P23) jet on.

7. Maintain the cask temperature and the jetting to_____for 1 hour after the cask temperature has reached 110°C.

8. When the drying operation is complete, turn off the P34 (P23) jet and turn off the steam to the cask coils.

9. Immediately turn on the air to the HAPO coil and set the air regulator at 75 psi. Do not open the air outlet from the coil.

10. Maintain the 75-psi pressure on the coil for 30 minutes. Observe the coil test-pressure gage in the cell. If the pressure goes above 30 psi, contact supervision.

11. If the coil test-pressure gage remains below 30 psi after 30 minutes, turn off the air pressure. Open the air outlet to relieve the pressure; then close the outlet.

12. Turn on chilled water to the coils and cool the cask to <20°C; then turn off the chilled water.

I. HAPO-II Cask Drying Procedure and Coil Leak Testing

1. Close the following valves from R11 to the cask:

   a. R11 to pump.

   b. Pump by-pass.

   c. Pump discharge.

2. Open the valve on the solution addition to line 1016.

3. Turn off the chilled water to the cask coil.

4. Adjust the temperature of the hot process water to 85°C.

5. Open the valve from cask to the P34 (P23) jet and the valve from the P34 (P23) jet to_____.

6. Turn on the P34 (P23) jet and jet to_____until jet temperature and_____off-gas indicate that the jet is blowing steam; then turn off the jet and close the valves.

7. Turn on hot process water (85°C) to the cask coil and open the steam discharge from the coil. Adjust hot process water flow rate to 5 gallons/minute.

8. Continue heating until the cask temperature reaches 80°C.

9. After the cask temperature has reached 80°C, turn off the hot process water and turn on the steam to the coil.

10. Heat the cask with steam until the cask temperature reaches 110°C.

11. When cask temperature reaches 110°C, continue steam heating. Open the valve from the cask to the P34 (P23) jet and the valve from the P34 (P23) jet to_____ only. Check that the valve on the solution addition to line 1016 is open.

12. Turn on the P34 (P23) jet and jet for 1 hour. Then do the following steps in sequence:

    a. Close the valve from the cask to the P34 (P23) jet.

    b. Turn off the P34 (P23) jet.

    c. Leave the valve from the P34 (P23) jet to_____open.

    d. Turn off the steam to the cask coil.

13. Immediately turn on the air to the cask coil and set the air regulator at 75 psi. Do not open the air outlet from the coil.

14. Maintain the 75-psi pressure on the coil for 30 minutes. Observe the coil test pressure gage in the cell. If the pressure goes above 30 psi, contact supervision.

15. If the coil test pressure gage remains below 30 psi after 30 minutes, turn off the air pressure. Open the air outlet to relieve the pressure; then close the outlet.

16. Before starting the cooling cycle (while steam heating and testing are being done) stabilize the temperature of the hot process water as follows:

    a. Set up a rotometer in Area 31, connecting it to the hot process water outlet and discharging to drain.

    b. Adjust the flow of hot process water to drain at 5 gallons/minute.

    c. Adjust the steam to the hot process water tank until a steady temperature of 50 to 55°C is obtained.

17. Open the hot process water to the cask coil and set the flow rate at 5 gallons/minute. Discharge the water through the steam outlet.

18. Check the temperature of the hot process water tank every 15 minutes and maintain temperature between 50 and 55°C.

19. Continue to cool until the cask temperature reaches 55°C.

20. Begin adjusting hot process water flow rate to maintain the cask temperature at 55 °C ± 2°C. Continue to control the hot process water tank temperature at 50 to 55°C.

21. Maintain the cask temperature at 55°C ± 2°C for 24 hours.

22. After the 24-hour aging period, turn off the hot process water to the cask coil.

23. Turn on chilled water to the cask coil and cool to 20°C; then turn off chilled water.

J. Disconnect of HAPO Cask

1. Check with supervision for permission to begin disconnect.

2. Check that all valves on the Cell 1 panelboard are closed except for the valve on the solution addition to line 1016.

3. Resurvey the work area over Cell 1-N. Unseal the plastic shield.

4. Open the cask vent valve and the valve from the cask to off-gas on the panelboard.

5. Attach the handling tool to the HAPO connector on the line to the P34 (P23) jet (this line is now on the "filtrate out" nozzle). Loosen the connector, remove it from the nozzle, and set the connector in the storage rack. Remove the handling tool.

6. Close the valve on the solution addition to line 1016.

7. Attach the handling tool to the HAPO connector on the line from R11 (this line is now on the "slurry in" nozzle). Loosen the connector in the storage rack. Remove the handling tool.

8. Remove all gaskets from the cupola.

9. Insert new gaskets onto the "filtrate out" and "slurry in" nozzles.

10. Set blank HAPO connectors onto the "filtrate out" and "slurry in" nozzles and tighten down as well as possible with the Allen wrench.

11. Insert the three outer caps over the blank HAPO connectors as follows:

    a. Insert a new O-ring gasket.

    b. Attach the outer cap to the handling tool and check that the hold-down bolts are in place.

    c. Insert the cap onto the process nozzle (over the HAPO connector).

    d. Turn the hold-down bolts with the long wrench until all are started in the threads. Begin tightening the bolts a few turns at a time; proceed from one bolt to the next and tighten them evenly. Manually tighten all bolts as firmly as possible.

12. When all three outer caps have been tightened, perform the pressure test as follows:

    a. Insert a hose into the cupola and add water to a point just over the outer cap bolts. Careful! Do not fill the cupola.

    b. Close the manual valve on the line from cask to off-gas. Leave the cask vent valve and the cask to off-gas valve on the panelboard open.

    c. Open the two valves on the air pressure to the vent system (these valves are designated HX-20111 and HX-20104) and set the pressure regulator (PIC-2011) at 90%.

    d. Close the two valves on the air pressure to the vent system (HX-20111 and HX-20104).

    e. Read the pressure on the vent line (PI-2019).

    f. Observe the water in the cupola for air bubbles and watch the pressure gage for any decrease. If either of these is observed, contact supervision.

    g. After caps have been determined to be leak-tight or the leaking caps have been identified, open the manual valve on the line from cask to off-gas.

    h. Set the air pressure regulator (PIC-2011) at 0.

    i. Open the two valves on the air pressure to the vent system (HX-20111 and HX-20104). Leave these valves open for 10 seconds; then close them.

    j. Attach the Snaptite suction line to the line from Cell 1-N to P12. Jet the water from the cupola to P12, and then turn off the jet.

k. Dry the cupola by swabbing. Place the swabs in a container in Cell 1-N.

l. If any leaks were found, the caps must be made leaktight before proceeding. Check with supervision.

m. When system is leaktight, proceed with final disconnect of cask.

n. Record final leak test pressure on Data Sheet No. 1.

13. Close the following valves:

a. Cask vent valve.

b. Manual valve on line from cask to off-gas.

c. Valve from cask to off-gas on panelboard.

14. Attach the Snaptite handling tool to the Snaptite on the cask vent nipple and remove it from the cask. Set the line in the storage rack.

15. Insert the blank Snaptite and attach it to the cask vent nipple. Remove the handling tool.

16. Insert the vent valve inner cap and position it over the vent valve.

17. Check that the large hold-down bolt is loose before going to step 18.

18. Insert the vent valve outer cap and position it over the inner cap.

19. Screw the outer cap down, and then tighten the hold-down bolt.

20. Open the air outlet from the cask coils to cell ventilation. Turn on the air to the cask coils and set the regulator at 75 psi.

21. Continue air purge of cask coil for 30 minutes; then set the regulator at 0, turn off the air supply, and turn off the air outlet.

22. Under plastic suit entry regulations, enter Cell 1-N. Survey and smear the cupola and the vent valve well. Decontaminate cupola to <100 mr/hr direct reading and <1 mr/hr smears.

NOTE: If remote decontamination is necessary, supervision will determine decontamination procedure.

23. Replace the vent valve well cover and tighten the screws.

24. Replace the cupola cover and tighten the nuts with the impact wrench.

25. Check that all valves on the Cell 1 panelboard are closed.

26. Disconnect the cooling coil lines and replace the blank flanges.

27. Disconnect the thermocouple and replace the cap.

28. Disconnect the coil leak test device and replace the cap.

29. Carefully remove the plastic wrapping from the cask. Survey and smear the cask. Decontaminate to low smear tolerance. Check with supervision for approval to remove the cask from Cell 1-N.

30. Remove the armor plate and the plastic shield.

31. Remove the cask from the cell. Decontaminate to shipping tolerance.

32. Record final smear results, survey results, and shipping date. Supervision and Health Physics will certify the cask as ready for shipment.

33. Turn in all data sheets for filing.

FISSION PRODUCTS DEVELOPMENT LABORATORY

HAPO CASK UNLOADING

DATA SHEET NO. 1

Run No._____

Cask No._____ Date Received_____

Radiation Reading_____ Smear Results_____

Connection

Cask Temp. (surface)_____°C Filter Temp._____°C

Cooling Procedure_____

_____

_____

_____

_____

Date Vented_____ Pressure_____

Unloading (Record Data on Sheet No. 2)

Time/Date Started_____ Time/Date Completed_____

Sample Data

First Slurry				Rinse			
Sample Code	Vol.	mc/ml ^{90}Sr	Total ^{90}Sr	Sample Code	Vol.	mc/ml ^{90}Sr	Total ^{90}Sr

Total ^{90}Sr Unloaded_____

Pressure Test - (Record Heat Cycle on Sheet No. 3) Test Pressure_____psi

Date Tested_____ Filter Temp. at Test Time_____°C

Shipping Data

Radiation Reading_____ Smear Results_____

Date Shipped_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

HAPO CASK UNLOADING

DATA SHEET NO. 2

Run No._____ HAPO Cask No._____

Date	Time	R11 Vol.	R11 Press.	P- Vol.	P- Press.	Cell 1 Elec.	Filter Temp.	Remarks

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

HAPO CASK UNLOADING

DATA SHEET NO. 3

Run No._____ HAPO Cask No._____

Heat Cycle for Coil Test

Date	Time	Filter Temp.	Heat Exchanger	Remarks

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

STRONTIUM-90 TITANATE PREPARATION

A. Preoperational Checks

1. Check the following in Cells 11 and 12:

   a. Manipulator arms are in good condition.

   b. Manipulator boots are in good condition.

   c. Hg vapor lights are operating.

   d. Cell ventilation is set betweel 1.0 and 1.5 inches of water.

   NOTE: The Cell 13 cell ventilation must be equal to or higher than that for Cell 12 (that is, more vacuum in Cell 13).

   e. Cell 11 furnace is in operating condition and has been tested (check with supervision for testing).

   f. Sumps are empty.

   g. Windows are clean.

   h. Airlocks are in operating condition.

2. Check that the following tools and equipment are in good operating condition in Cell 11:

IMPORTANT NOTE: Since much of the equipment used in the ^{90}Sr process is similar to that used in the ^{137}Cs process, care must be taken to assure that the equipment does not get mixed up. Except where noted below, all equipment used in the ^{90}Sr process is marked ^{90}Sr; the ^{137}Cs equipment is unmarked.

   a. Two filter shells (unmarked) with covers and V-band clamps.

   b. Safety bottle (4-liter vacuum flask) with stopper.

   c. Two long spatulas.

   d. Filter lifting tool.

   e. Filter handling tool (3-prong).

   f. Filter stand (beaker on 45° base).

   g. Pan balance with weights.

   h. Triple beam balance.

i. Two calorimeter cups.

j. Calorimeter cup lifting tool.

k. Two 1-liter and two 2-liter stainless steel beakers.

l. Scoops and brushes.

m. Hose clamps and hose clamp wrench.

n. Tygon lines for filters, filter gaskets, and pads.

B. Chemical Operation - Setup

1. Check that the feed preparation in Cell 4 is completed and that the solution in P34 has been sampled.

2. Supervision will determine the feed batch data to be entered on the _____Sr11 Preparation Data Sheet.

3. Check that all valves and services on the Cell 11 panelboard are closed except the R111 off-gas.

4. Check that P511, H511, and R111 are empty.

5. Assemble two filters according to the "Filter Buildup Procedure."

NOTE: Check that the filter thimbles are marked "^{90}Sr_____." The secondary filter from a previous run may be used if it is in good condition.

6. Using Tygon tubing, connect the outlet from the P511 bottom discharge to the inlet to the primary filter. Connect the outlet from the primary filter to the inlet of the secondary filter. Connect the outlet from the secondary filter to the inlet to the safety bottle. Connect the outlet from the safety bottle to the inlet to R111.

7. Close the bottom discharge valve on P511.

8. Close the R111 off-gas valve, turn on the cooling water to the R111 vacuum steam jet condenser, and turn on the R111 vacuum steam jet.

9. Observe the R111 vacuum gage. If the gage reaches_____feet of water and holds steady, the system is leak-tight and the operation can proceed. If the system is not leak-tight, all leaks must be corrected before proceeding.

10. When the system is leak-tight, turn off the R111 vacuum steam jet.

11. Open the R111 off-gas valve, then close it immediately. Repeat this operation until the R111 vacuum is bled off; then leave the R111 off-gas valve open.

12. Turn off the cooling water to the R111 vacuum steam jet condenser.

13. Check that the following equipment is set up on top of the Cell 11 instrument cubicle (in crane bay):

    a. 50-liter polyethylene vessel with bottom discharge line and agitator.

    b. Waring blender and blender base.

    c. Balances.

14. Contact supervision for any special cleaning instructions pertaining to Cell 11.

D. Chemical Operation - Precipitation

1. Contact supervision for approval to begin operations. Supervision will designate batch code and enter starting data on the Preparation Data Sheet.

2. Set up communications between panelboard 2 and Cell 11.

3. Open the valve from P34 to H511 (on the Cell 11 panelboard).

4. Turn on the jet from P34 to H511 and jet_____gallons of solution from P34 to H511; then turn off the jet. (Check with supervision for volume to be transferred.) Close the valve from P34 to H511.

5. Check that P511 has at least 6 inches of off-gas and recheck that the P511 bottom discharge valve is closed.

6. Open the valve from H511 to P511.

7. Turn on the jet from H511 to P511 and jet the solution from H511 to P511. Do not exceed 80% liquid level in P511.

8. When H511 is empty (or P511 reads 80% liquid level), turn off the jet from H511 to P511 and close the valve from H511 to P511.

9. If necessary, evaporate P511 as in Steps 13 through 16 below; then repeat Steps 6, 7, and 8 until H511 is empty.

10. Add 8 liters of demineralized water to H511.

11. Open the valve from H511 to P511 and turn on the jet. When H511 is empty, turn off the jet and close the valve.

12. Repeat Steps 10 and 11 once.

13. Turn on the P511 agitator.

14. Turn on the steam to P511 and begin evaporation.

15. Evaporate P511 to a volume of 45 liters (assume specific gravity to be 1.2); then turn off the steam to P511.

16. Turn on the chilled water to P511 and cool P511 to 40°C; turn off the chilled water.

17. Agitate P511 for 15 minutes, then pull three samples. Codes _____Sr11-F1, _____Sr11-F2, _____Sr11-F3. Request acid normality and ^{90}Sr rush.

18. When acid normality is known, check with supervision for the volume of NH_4OH to be added in Step 19.

19. Add_____milliliters of concentrated NH_4OH to P511 and rinse the solution addition line with 500 milliliters of demineralized water.

20. Agitate P511 for 10 minutes after the NH_4OH addition; then pull a sample from P511 and check the pH.

21. If the pH of the solution in P511 is less than 3.0, do the operations below until the pH is between 3.0 and 5.0:

    a. Add 200 milliliters of concentrated NH_4OH to P511 and rinse the solution addition line with 500 milliliters of demineralized water.

    b. Agitate P511 for 10 minutes and recheck pH.

    c. Repeat a and b until pH is 3.0 to 5.0.

    d. Check sample for precipitate.

22. When pH is between 3.0 and 5.0 and no precipitate is visible, proceed. If the pH goes above 5.0 or if any precipitate is noted, contact supervision.

23. Hold the solution in P511 until the ^{90}Sr content is known from analytical results. Control the temperature of P511 below 50°C.

24. Supervision will make the necessary calculations. Enter the appropriate data on the Preparation Data Sheet.

25. Make up the precipitating solutions as follows:

    a. Add 18 liters of demineralized water to the polyethylene vessel on the Cell 11 cubicle and turn on the agitator.

    b. Slurry_____grams of TiO_2 in a Waring blender approximately 50% full of demineralized water. Blend for 2 minutes and then add the contents of the blender to the 18 liters of water in the polyethylene vessel. Rinse the blender into the vessel, using 1 liter of demineralized water (check with supervision for TiO_2 weight).

c. Add 5130 grams of ammonium carbonate and 1 liter of concentrated NH_4OH to the polyethylene vessel and maintain agitation.

26. Place the bottom discharge line from the polyethylene vessel into the solution addition funnel to P511.

27. Open the solution addition funnel to P511; then open the clamp on the bottom discharge line from the polyethylene vessel and add the slurry to P511. When the slurry level in the polyethylene vessel drops below the agitator, turn off the agitator.

28. Rinse the solution addition line to P511 with 2 liters of demineralized water; then close the solution addition valve and close the clamp on the polyethylene vessel discharge.

29. Turn on the steam to P511 and heat to 72°C.

NOTE: Watch the P511 off-gas. If it goes below 4 inches, reduce the heating rate.

30. Digest P511 at 70 to 72°C for 30 minutes.

31. While P511 is digesting, add 19 liters of demineralized water, 1 liter of concentrated NH_4OH, and 1000 grams of ammonium carbonate to the polyethylene vessel and turn on the agitator.

32. After the 30-minute digestion (while P511 is still at 70 to 72°C), open the solution addition valve to P511 and add the solution from the polyethylene vessel to P511 by opening the clamp. When the level in the vessel drops below the agitator, turn off the agitator.

33. Rinse the solution addition line with 2 liters of demineralized water; then close the solution addition valve and clamp.

34. Turn off the steam to P511. Turn on the chilled water to P511 and cool P511 to 30°C over a period of 30 minutes by manual control of the chilled water valve.

35. Sample P511 and check the pH. If the pH is below 8.0, contact supervision.

E. Chemical Operation - Filtration

1. Maintain P511 temperature at 30°C ± 2°C.

2. Open the bottom discharge valve from P511 and allow solution to flow into the filter.

3. Close the R111 off-gas valve.

4. Open the water to the R111 vacuum steam jet condenser.

5. Turn on the R111 vacuum steam jet and begin filtration. Watch for leaks in the system and for any evidence of precipitate going into the safety bottle. If either occurs, close the bottom discharge valve from P511, turn off the R111 vacuum steam jet, open the R111 off-gas valve, and contact supervision.

6. When the P511 liquid level reaches 15%, turn off the P511 agitator.

7. While the filtration is being done, make up 5 liters of rinse solution. Makeup: Dissolve 575 grams of ammonium carbonate in demineralized water, add 200 milliliters of concentrated NH_4OH, and dilute to 5 liters with demineralized water.

8. When P511 is empty, open the valve on the water to the P511 spray ring, leave it open for 10 seconds, then close it and immediately add 1 liter of rinse solution to P511.

9. Allow P511 to pull empty (watch the Tygon line between the P511 bottom discharge and the primary filter).

10. Repeat Steps 8 and 9 four more times.

11. Continue drying with vacuum for 30 minutes after the last rinse has been done.

12. Turn off the R111 vacuum steam jet.

13. Open the R111 off-gas valve, then close it immediately. Repeat this operation until the R111 vacuum has been bled off.

14. Turn off the water to the R111 vacuum steam jet condenser.

15. The precipitate and ceramic filter are now ready to be removed from the filter shell. Check with supervision.

16. Loosen the V-band clamp on the primary filter.

17. Remove the top of the primary filter.

NOTE: There will be a considerable evolution of steam at this point.

18. If any precipitate adheres to the top section of the filter shell (stainless steel) or the gaskets, scrape it off into the filter thimble. Do not scrape down the sides of the filter thimble at this point. If the filter thimble is broken, contact supervision.

19. Using the filter lifting tool, raise the filter thimble out of the sheel and set it upright in a stainless steel beaker.

20. Remove the top from the secondary filter and remove the thimble. Inspect for precipitate. If the filter is in good condition, it may be re-used.

21. Close the bottom discharge valve on P511.

22. Remove all gaskets and pads from the filter shells and place them in a waste can. If any precipitate aheres to this material, scrape it off into the filter thimble containing the bulk precipitate. Gaskets and pads cannot be re-used.

23. Contact supervision for R111 sampling instructions.

F. Chemical Operation - Calcination

1. Allow the precipitate to remain in the filter thimble until no more steam is evolved. If there is still steam after 2 hours, contact supervision.

2. Carefully scrape down the sides of the filter thimble with a spatula. Slowly stir the precipitate and attempt to break up lumps. If more steam evolution is noted, allow the precipitate to dry for an additional 30 minutes and then stir again.

3. Carefully lift the filter thimble out of the beaker and lay it down in a horizontal position on the cell floor.

4. Insert the filter thimble into the 3-prong filter handling tool so that the open end of the thimble faces the apex of the tool.

5. Open the furnace door.

6. Carefully lift the filter thimble and insert it into the furnace. Check that the thimble is clear of the door, then remove the handling tool.

NOTE: More than one batch may be calcined at a time. Check with supervision.

7. Close the furnace door.

8. Install the 1100°C template on the programmer.

9. Check the temperature indicator and recorder. It may be necessary to touch the thermocouple connectors in the cell to assure good connection.

10. Turn on the programmer. (Set the microswitch contact arm so that it will not turn off the programmer.)

11. Turn on the furnace.

12. Check the furnace closely during the first 4 hours of operation, then record readings each hour.

13. The temperature should increase from cell temperature to 1100°C at approximately 100° per hour. Hold at 1100°C for 4 hours, then cool to 800°C at approximately 100°C per hour.

14. When furnace reaches 800°C on the cooling cycle, turn off the furnace power.

15. Allow the furnace to cool to 300°C; then open the furnace door approximately 1 inch.

16. Open the furnace door approximately 1 inch each hour until the temperature reaches 200°C; then open the door completely.

17. Insert the handling tool and withdraw the thimble from the furnace. Set the thimble into the filter stand.

18. Stir the powder with a spatula and attempt to break up lumps.

19. Tare a stainless steel beaker; then transfer the powder from the filter thimble into the tared beaker and determine the total powder weight.

20. Prepare two calorimeter samples and assay on the Cell 12 calorimeter.

21. When the calorimetry is completed, return the calorimeter cups to Cell 11 and empty the contents of the cups back into the powder batch.

22. Record calorimeter results on Preparation Data Sheet.

23. Check with supervision for instructions. Supervision may direct additional calorimetric measurements, temporary storage for the powder, or storage in the well.

24. Turn in completed data sheets (run sheet, furnace record, calorimeter data) to supervision.

FISSION PRODUCTS DEVELOPMENT LABORATORY

STRONTIUM TITANATE PREPARATION

DATA SHEET

Batch Number _____ Time/Date Started _____

Status (before operations)

Feed batch identification (from P34) _____

$^{89}Sr/^{90}Sr$ ratio _____ (as of _____)

Total moles alkaline earth per kc ^{90}Sr _____

P511 samples:

Anal.	-F1	-F2	-F3	Avg.
^{90}Sr mc/ml				
Vol P511				
Total ^{90}Sr				

Operation

Weight TiO_2 added _____ g Initial pH _____ Final pH _____

Filtration times: Started _____ Completed _____

Filtrate volume _____ Total curies ^{90}Sr in filtrate _____

Calcination times: Started _____ Completed _____

Product Data

Total weight product _____ g

Calorimeter results: 1st check _____ c/g; 2nd check _____ c/g

Total ^{90}Sr curies _____ Total ^{90}Sr watts _____

Product disposition _____

Remarks: _____

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

STRONTIUM-90 TITANATE HOT-PRESS OPERATION

NOTE: This procedure is also used for hot pressing of ^{90}SrO pellets, but special powder handling is required. Supervision will issue special instructions for ^{90}SrO work.

A. Cell Preparation

1. Check that there is no flammable debris in Cell 12.

2. Adjust cell ventilation pressure to a minimum of -1.0 inch water.

3. Inspect manipulators for proper operation. Inspect both boots for holes.

4. Place furnace in horizontal position.

B. Equipment

1. Insert the following equipment into Cell 12 (or check that equipment is already in cell):

   a. Graphite die and punches (check with supervision for size).

   b. Graphite collar and collar punch.

   c. Die handling tool.

   d. Collar handling tool.

   e. Pt, Pt-10% Rh thermocouples; thermocouple sheaths (2 each).

   f. Mica disks, mirror.

   g. Powder handling tools (spatula, funnel, etc.).

   h. Ejection punch and plates for ejection press.

   i. Pre-press punch.

   j. Die liner or screen inserts if required.

C. Auxiliary Services for Furnace

1. Turn on the process cooling water to the hot press by opening the valve on the face of the cell.

NOTE: Observe the liquid level in H209 before turning on the process water to the hot press and generator. Pump H209 to M.H.-209 when the H209 level reaches 60%.

2. Adjust the flow with this valve to a minimum of 40% on the rotameter.

3. Observe the in-cell process water hose and Snaptites on the furnace assembly for leaks. Contact supervision if any leaks are noted.

4. Open the argon supply manual valve on the face of Cell 12.

5. Open the argon cylinder manual and regulator valve. Adjust the flow to a rate of 50 cubic feet per hour with the regulator valve on the face of Cell 12.

D. Die Loading (Supervision Must Be Present)

1. Place the pre-press bottom plate in ejector assembly.

2. Place the bottom punch in the graphite die body and set die into the ejector press, using the die handling tool.

3. If liner is required, put liner into die.

4. Transfer_____grams of powder from the powder handling cup or beaker to the graphite die body. Insert the pre-press punch in the die.

5. Apply_____psig pressure to the pre-pressed pellet. Release pressure and remove pre-press punch.

NOTE: Close the hot-press manual valve for the hydraulic system in the cell and open the ejector press manual valve before applying pressure.

6. If screen inserts are used, place one insert on top of powder.

7. Continue adding powder in ~10% increments and reapplying_____psig pressure until all the powder is in the graphite die body. (Place inserts as directed by supervision.)

8. If liner is used, place the liner lid on the pre-pressed pellets; fold down the liner edges.

9. Insert the top punch into the die.

E. Furnace Loading (Supervision Must Be Present)

1. Lift the graphite die body from the ejector press, using the die handling tool. Remove any tape that is attached to the die body.

2. Transfer the die body to the rear of the furnace.

3. Lift the collar with the collar handling tool and insert into the furnace.

4. Insert the graphite collar punch into the collar.

5. Insert the thermocouple sheaths and thermocouples into the collar holes. Check that the bottom of the thermocouple sheath is in contact with the die body.

6. Connect the thermocouples to the connectors located on the furnace top plate. Check the temperature recorder for proper thermocouple response.

7. Connect the air hoist hook to the lifting eye on the left rear of the furnace.

8. Engage the safety bar on the air hoist hook.

9. Lift the furnace to its vertical position. Observe carefully that the furnace lead wires, cooling water lines, and thermocouple lead wires are positioning themselves properly during the lifting operation.

10. Remove the air hoist hook from the furnace lifting eye.

11. Place two mica insulating disks on the collar punch.

12. Place the steel punch on top of the collar insulating disk.

13. Place two mica insulating disks on top of the steel punch.

14. Open the hot-press manual valve to the hydraulic system in the cell and close the ejector-press manual valve before applying pressure.

15. Lower the furnace hydraulic ram and check alignment of the ram, steel punch, and collar punch with a mirror.

NOTE: Do not allow the hydraulic ram to make contact with the steel punch.

16. Raise the hydraulic ram.

F. R.F. Generator Operation

NOTE: Manipulators are NOT to be moved to the rear of the cell when generator is under load.

1. Plug the generator electrical connector into the 440-volt welding outlet and turn on the welding outlet switch.

2. Preset the generator autotransformer tap switch to the No. 3 position.

3. Preset 200-kva (kilovar) capacitance, using both the 100-kva switches located on the top of the generator.

4. Turn on the generator motor by depressing the start switch located at the bottom of the generator.

5. Adjust the power output control knob to zero.

6. After generator motor is up to full speed, depress the field control switch located on the top left side of the cubicle.

7. Depress the load switch located on the top right side of the cubicle.

8. Slowly turn the power output control knob clockwise until a load of between 10 and 15 kilowatts is noted. Supervision will determine the power to be applied.

NOTE: In adjusting to between 10 and 15 kilowatts, do not allow the generator output to exceed 440 volts or 45.5 amperes. The kilovar meter reading should be between 1 and 5 kva lead. If the above conditions cannot be obtained, contact supervision.

9. Record the following data every 15 minutes:

   a. Setting of power output control knob.

   b. Furnace temperature.

   c. Power output.

   d. Generator output voltage.

   e. Generator output amperage.

   f. Pellet temperature.

   g. Water flow.

   h. Cell temperature.

   i. Argon flow.

   j. Argon cylinder pressure.

NOTE: The following heating and pressing cycles are subject to change and should be noted carefully before each run.

10. Heat the furnace to_____°C.

NOTE: For a floating die body, apply and maintain 500 psig during the heating and holding cycles.

11. Hold the furnace at_____°C for_____minutes.

12. Lower furnace temperature to_____°C.

13. Apply_____psi on the gage. Apply this pressure at a rate of ~300 psig per minute.

14. Maintain_____psi on the gage for_____minutes.

15. Raise the press ram slowly. Depressurize at a rate of ~300 psig per minute.

16. Turn the furnace off by turning the power output control knob to zero. Depress the stop switches on the load, field control, and generator motor in the order listed.

17. Allow the furnace to cool to a temperature of_____°C. Turn off argon and water to the furnace when temperature reaches_____°C.

G. Removal of Die from Furnace

1. After the furnace has cooled to_____°C, remove the steel punch and mica disks from atop the collar punch.

2. Attach the air hoist hook to the furnace lifting eye. Engage the air hoist safety bar.

3. Lower furnace to its horizontal position.

4. Disconnect thermocouples and remove thermocouples and sheaths from collar.

5. Remove collar and collar punch from furnace.

6. Clear out area in front of furnace.

7. Remove die from furnace.

H. Pellet Ejection (Supervision Must Be Present)

1. If the pellet is to be loaded into a liner immediately, set up liner. If the pellet is to be stored, check storage location. Also check that balances, pellet handling tool, and measuring devices are in cell and ready for use.

2. Remove the pre-press bottom plate from the ejector press. Set the ejector die guide in the ejector press. Check that the proper size adapter post is in place.

3. Invert the die body and place it into the guide, with the top punch down.

4. Position the ejector collar on top of the die body; make sure it is centered.

5. Close the hot-press hydraulic manual valve and open the ejector-press manual valve.

6. Slowly open the hydraulic control valve until the ram contacts the ejector collar, then increase pressure until the die body begins to move.

NOTE: If the pressure reaches a value equal to one-half of the final hot-pressing pressure (Step F.14), hold. Supervision will determine further steps to be taken.

7. When the die body begins to move, maintain just enough pressure to continue movement. Observe the travel of the punch and pellet. When the pellet is completely ejected, release the pressure and raise the ram.

8. Carefully remove the ejector collar; do not allow the collar to touch the punch or the pellet.

9. Carefully remove the punch from the pellet.

10. Transfer the pellet to the balances; weigh and record weight. Measure the pellet height and record.

11. Transfer the pellet to the liner or storage as instructed by supervision.

12. Remove the die guide from the ejector press and insert the bottom ejection plate.

13. Set the die body into the ejection press. Using a steel punch, extrude the punch from the bottom of the die.

NOTE: The die body and punches will be inspected by supervision, who will determine whether they can be re-used.

FISSION PRODUCTS DEVELOPMENT LABORATORY

HOT PRESS DATA

Run Description _____

Material _____ Wt _____ Wt Pt _____ Die Dia. _____ Watts _____ Curies _____

Date	Time	Furnace Temp. °C	Power Output	Gen. Output		Water Flow	Argon		Press Gage Pressure	Cell Temp. °C	Remarks
				Volts	Amps		Flow	Press.			

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

PELLET DATA SHEET

Customer: _____

Compound: _____ Batch No. _____

Weight of Powder: _____ Can No. _____

Activity: _____ c/gm _____ curies _____ watts

_____ w/gm

Liner Material _____ Liner Wt. _____

Pellet Wt. _____ Pellet Height _____

Pellet Diameter _____ Temperature of Run _____

Pressure (gage) _____ Pressure (applied) _____

Pellet Approved by Supervision _____ _____

Signature Date

Calorimetry:	Run #1 Date:	Run #2 Date:	Run #3 Date:
Wt. Sample			
Mr			
Calorimeter Constants			
Watts/gm			
c/gm			

Remarks (Use Reverse Side if Necessary):

FISSION PRODUCTS DEVELOPMENT LABORATORY

STRONTIUM-90 TITANATE PELLET PRESSING/SINTERING

NOTE: Each $^{90}SrTiO_3$ pellet preparation campaign is a special project and special procedures may be issued. The procedures below are generalized.

A. Preliminary Setups

1. In Cell 12, check that the following tools and equipment are in good condition:

   a. Pellet press (check for operation by turning on pump motor and turning control valve to raise and lower press). Check pump oil level.

   b. Extrusion jack.

   c. Storage can wrench and vise.

   d. Spatulas and probes as needed.

   e. Balances and weighing pans, weights.

   f. Dial gage with jo-blocks.

   g. Pellet scoop and push tool.

   h. Sintering tray transfer scoop.

2. Check that manipulator's arms and boots are in good condition.

3. Check with supervision for special instructions and equipment and insert the equipment. Equipment may include (but is not limited to) the following:

   a. Die sets (body, punches, shims, guides).

   b. "Go-no" gage.

   c. Sintering trays with ZrO_2 granules.

   d. Stearic acid lubricant.

   e. Die cleaning tools (matched to die).

   f. Die handling tools.

   g. Die loading funnel and brushes.

h. Calibrated powder scoop or weighing beaker.

i. Extrusion punches and rings.

NOTE: Supervision will issue special data sheets as required and will designate the powder batch(es) to be used.

4. Transfer the designated powder to Cell 12, open the cans, and transfer the powder into storage beakers. Check with supervision for powder data (may request calorimetry).

5. Check with supervision for loading procedure.

   a. If large die (>1-cm pellets) is to be used, follow the procedure outlined in section B and omit section C.

   b. If small die (<1-cm pellets) is to be used, follow procedure outlined in section C.

B. Large-Die Loading

1. Lightly lubricate the die body, short punch, and long punch (but not the shims).

2. Lift the die body to the press and engage the ears of the die body in the slots of the suspension arms; leave die body suspended.

3. Center the guide ring on the press base by pushing the ring against the V-slot on the base; then place the short punch into the hole in the guide ring (notched end down), and place one shim on top.

4. Turn on the hydraulic pump.

5. Slowly raise the press by turning the hydraulic control valve clockwise. As the press rises, guide the die body so as to insert the short punch into it. Continue turning the valve until the die body touches the guide ring; then stop the press.

6. Disconnect the suspension arms from the die.

7. Slowly turn the control valve counterclockwise to lower the press; turn off the pump.

8. Place the funnel into the die.

9. Measure the proper amount of the designated powder by (a) individual weighing or (b) volume measurement; supervision determines which method to use.

a. If individual weighing is to be done, weigh the powder out into the weighing beaker by standard procedure.

b. If volume measurement is to be done, weigh the specified amount of powder sufficient to make several pellets; then use the calibrated spatula to measure out powder for each pellet. When using the calibrated spatula, be sure it is level full. Use the flat side of a spatula to scrap excess powder back into the beaker.

10. Transfer the powder from the weighing beaker (or calibrated spatula) into the funnel.

11. Check the beaker and funnel to be sure that all powder has been transferred into the die; then remove the funnel.

12. Insert one shim into the die.

13. Insert the long punch into the die (notched end up).

14. Set the long ejector on top of the punch, and be sure that the guide tip inserts into the notch on the end of the long punch.

C. Small-Die Loading

1. Lightly lubricate the die body and punches.

2. Place the short punch in the die body.

3. Set the die (punch down) in the handling clip.

4. Check with supervision for the number of spacers to be placed on the press. Center the spacers on the press base.

5. Set the funnel into the die.

6. Measure the proper amount of the designated powder as instructed by supervision. (See step B.9 above.)

7. Transfer the powder from the weighing beaker (or calibrated spatula) into the funnel.

8. Check the beaker and funnel to be sure that all powder has been transferred into the die; then remove the funnel.

9. Insert the long punch into the die.

10. Lift the die in the handling clip and set the clip in the center of the top spacer on the press base.

D. Pressing

1. Check with supervision for the required pressures.

2. Turn on the hydraulic pump.

3. Slowly turning the hydraulic control valve clockwise, raise the press until the top of the die assembly touches the top plate of the press.

4. When the pressure gage begins to respond, continue slowly turning the control valve until the required pressure is reached.

5. When the required pressure is reached, stop turning control valve (do not turn valve back). Hold pressure for 5 minutes.

6. NOTE: Adjust pressure back to required pressure if it falls off during the 5-minute hold.

7. NOTE: For small (<1 cm) pellets, omit the remainder of this section and proceed immediately to Section F below. For large (>1 cm) pellets, complete the remainder of this section; then proceed immediately to Section E.

8. Engage the suspension arms onto the die body.

9. Release the pressure by turning the hydraulic control valve counterclockwise to lower the press.

10. NOTE: Hold the long ejector during this operation; remove it as it releases from the assembly.

11. The die body will now be suspended on the suspension arms. Remove the guide ring from the press base.

12. Invert the die body so that the long punch is down.

13. Place the short ejector in the center of the press base.

14. Turn the hydraulic control valve slowly clockwise and continue turning until the guide tip on the short ejector inserts on the slot in the end of the long punch (adjust with manipulators if necessary).

15. Set the long ejector on top of the short punch and engage the guide tip.

16. Slowly turn the control valve to raise the press until a pressure of ~100 psig shows on the gage.

17. Disconnect the suspension arms from the die.

18. Turn the control valve clockwise until the required second pressure is reached.

19. When the required second pressure is reached, stop turning the control valve (do not turn valve back). Hold pressure for 5 minutes.

20. NOTE: Adjust pressure back to required pressure if it falls off during the 5-minute hold.

21. Proceed immediately to step E.

E. Large-Die Extrusion

1. Slowly turn the control valve counterclockwise until the pressure is ~200 psig.

2. Insert the extrusion jack onto the die so that the bottom of the jack rests on the shoulder of the die body. Hold jack in position.

3. Operate the jack handle until the ram of the jack contacts the top plate of the press.

4. Continue operating the jack until the die body is pushed to the press base, leaving the pellet and shims exposed.

5. Hold the jack with manipulators and slowly lower the press. When jack is free, remove it. Turn off the hydraulic pump.

6. Remove the long ejector.

7. NOTE: Pick the ejector straight up until it is clear, then move it sideways.

8. Hold the pellet scoop so that the bottom shim will slide onto it. Using the push tool, carefully slide the pellet and shims onto the scoop.

9. Set the scoop onto the floor and remove the top shim.

10. Hold the scoop just over the surface of the ZrO_2 granules on the sintering tray; carefully slide the pellet off the shim and onto the ZrO_2.

11. Inspect the pellet for flaws (chipping, cracks, etc.) and contact supervision if any are seen.

12. Clean the die body with the cleaning rod. If more pellets are to be made, proceed. If die is to be stored, contact supervision for instructions as to lubricating and storing the die assembly.

F. Small-Die Extrusion

1. Turn the hydraulic control valve counterclockwise and lower the press.

2. Set the die, in the handling clip, onto the floor.

3. Remove the die from the clip, invert the die, and replace it in the clip with the long punch down.

4. Replace the die, in the clip, on the press and center it on the spacer.

5. Hold the slotted extrusion tool on the shoulder of the die. Turn the hydraulic control valve clockwise to raise the press (continue holding extrusion tool) until the top of the extrusion tool contacts the top plate of the press.

6. Slowly increase the pressure and slide the die body down, exposing the short punch and the pellet. Stop increasing pressure when die body is down.

7. Hold the extrusion tool with manipulators and turn the control handle counterclockwise to lower the press. Turn off pump motor.

8. Remove the extrusion tool by lifting it straight up.

9. Lift the short punch off the pellet.

10. Hold the pellet scoop so that the pellet will slide onto it. Using the push tool, carefully slide the pellet onto the scoop.

11. Hold the scoop just over the surface of the ZrO_2 granules on the sintering tray; carefully slide the pellet off the scoop and onto the ZrO_2.

12. Inspect the pellet for flaws (chipping, cracks, etc.) and contact supervision if any are seen.

13. Clean the die body with the cleaning rod. If more pellets are to be made, proceed. If die is to be stored, contact supervision for instructions as to lubricating and storing the die assembly.

G. Sintering

1. When a full furnace load (three trays) of pellets has been pressed or when a pellet run has been completed, place each sintering tray into a transfer scoop and transfer the trays to Cell 11.

2. Open the furnace door.

3. Keeping the tray level, lift each scoop to the furnace door and push the sintering tray into the furnace; then remove the scoop.

4. Arrange the sintering trays in the furnace as follows:

No. of Trays	Position
1	Centered in furnace.
2	Side by side, centered in front-to-back direction.
3	Two side by side, pushed to rear; one crosswise in the front of the furnace.

5. Close the furnace door.

6. Install the 1400°C template on the programmer.

NOTE: The 1400°C sintering temperature is standard, but supervision may direct different sintering cycles.

7. Check the temperature indicator and recorder. It may be necessary to touch the thermocouple connectors in the cell to assure good connection.

8. Turn on the programmer. (Set the microswitch contact arm so that it will not turn the programmer off.)

9. Turn the furnace on.

10. Check the furnace closely during the first 4 hours of operation; then record readings each hour.

NOTE: The temperature should increase from cell temperature to 1400°C at ~100°C per hour; hold at 1400°C for 8 hours, then cool to 800°C at ~100°C per hour.

11. When the furnace reaches 800°C on the cooling cycle, turn off the furnace power.

12. Allow the furnace to cool to 300°C; then open the furnace door ~1 inch.

13. Open the furnace door ~1 inch each hour until the temperature reaches 200°C; then open the furnace completely.

14. Slide each tray out of the furnace and onto a transfer scoop.

15. Check with supervision for measurement and storage procedures.

H. Pellet Measurements (Cell 12)

1. General

   a. Pellets of weights up to 100 g may be handled directly with manipulators. Large pellets will be handled with special tongs.

   b. Do not let pellets touch plastic, paper, water, wood, or rubber (except manipulator grips). If a pellet accidentally comes in contact with any of these materials, contact supervision.

   c. Important: Pellets cannot be identified except by their location in the cell. Handling and storage procedures will be issued with each project and will include methods for location of pellets. Be sure to show the status of all pellets in the cell at shift changes.

   d. During pellet handling, report any defects that may be observed to supervision.

2. Weighing

   a. Check that balances are operating properly.

   b. Tare a weighing pan.

   c. Lift the pellet off the ZrO_2 and inspect the pellet to see if any ZrO_2 granules are stuck to it. Brush the pellet if necessary.

   d. Place the pellet into the weighing pan.

   e. Weigh the pellet and pan. Subtract the tare weight; record the pellet weight on the data sheet.

   f. NOTE: In cases where small pellets are weighed as a group, record the number of pellets and their total weight.

   g. Return the pellet to the ZrO_2.

3. Height-Diameter Measurements

   a. Set the proper number of calibrated jo-blocks on the base of the dial gage while holding the dial gage plunger up. (Number of blocks is based on estimated pellet height; check with supervision.)

b. Let the dial gage plunger come down to touch the top block. Release plunger and take reading. Lift the plunger and again lower it and release; reading should remain the same. (If it does not, contact supervision.)

c. Determine the "zero" height by adding the heights of the jo-blocks to the reading on the dial gage.

d. Lift the pellet off the ZrO_2 and inspect it; brush ZrO_2 off the pellet if necessary.

e. Raise the dial plunger and set the pellet under the plunger (on top of jo-block).

f. Release the plunger and let it contact the pellet.

g. Slowly slide the pellet around on the block while watching the gage. Continue until a maximum reading is obtained on the gage. (Gage must be on scale; otherwise more jo-blocks are needed.)

h. Add the maximum reading on the dial gage to the total height of the jo-blocks.

i. Subtract the "zero" measurement determined in (c) above from the value determined in (h) to determine the pellet height. Record pellet height on the data sheet.

j. Measure the diameter as in steps a through i, except that the pellet is rotated under the plunger while being held with manipulators. Record diameter on the data sheet.

4. Other Measurements

Check with supervision for instructions on other measurements needed; these may include the following:

a. Calorimetry (on some or all pellets).

b. "Go-no" gage checks (diameter and/or height).

c. Packing factor determinations.

d. Surface temperature measurements.

FISSION PRODUCTS DEVELOPMENT LABORATORY

YTTRIUM EXTRACTION

NOTE: The starting point for this operation is after a batch of rare earth feed concentrate solution has been transferred to H89.

A. Chemical Makeup

1. 3 gallons of 19 M C.P. NaOH.

2. 26.4 liters of Amsco.

3. 6.6 liters of D2EHPA.

4. 3.0 gallons of 1 M HNO_3.

B. Operation

1. Check that all jets and valves are closed on Tank N-19 and that tank is ready to receive solution from H-89. Jet solution from H-89 to N-19.

2. Cool N-19 to 30°C.

3. Turn off agitator and record volume on Batch Data Sheet. Assume specific gravity of 1.1.

4. Turn on agitator and sample. Code _____Y1.

5. Add 2.0 gallons of 19 M C.P. NaOH solution while controlling temperature below 50°C. Flush addition line with 2 liters of water.

6. Agitate N-19 for 10 minutes, and sample solution for acid normality. Make titration in Cell 19. Record acid normality on Batch Data Sheet.

7. Adjust acid normality to 1 N ± 0.1 N free acid by adding 500 milliliters of 19 M C.P. NaOH or 500 milliliters of 70% HNO_3 for each 0.1 above or below 1.0 N. Hold temperature at 20 to 25°C with water on jacket. Flush addition line with 2.0 liters of water after each NaOH addition.

8. Add 6.6 liters of D2EHPA to N-19.

9. Add 26.4 liters of Amsco to N-19.

10. Agitate N-19 for 10 minutes at 20 to 25°C.

11. Turn off agitator and let N-19 settle for 10 minutes.

12. Check R-79 to see that all jets are off and tank has been rinsed and is ready to receive solution from N-19.

13. Open valve from N-19 to R-79 and separate aqueous phase from the organic phase by airlift flow of aqueous to R-79. Aqueous phase contains all rare earths except yttrium. Set air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and the valve from N-19 to R-79 will automatically close.

14. When the alarm sounds, cut off hand switch.

15. Check N-29 to see that all valves and jets are closed and tank is empty.

16. Jet the aqueous solution containing cerium and promethium from R-79 to N-29.

17. Add 1.5-gallon portion of 1 M HNO_3 to N-19 and let settle for 3 minutes.

18. Open valve from N-19 to R-79 and separate aqueous phase from the organic phase by setting air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and the valve from N-19 to R-79 will automatically close.

19. When the alarm sounds, cut off hand switch.

20. Check N-29 to see that all valves and jets are closed and tank is ready to receive the HNO_3 rinse.

21. Jet the aqueous solution from R-79 to N-29.

22. Add 1.5 gallons of 1 M HNO_3 to N-19 and let settle for 3 minutes.

23. Open valve from N-19 to R-79 and separate aqueous phase from the organic phase by setting air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and the valve from N-19 to R-79 will automatically close.

24. When the alarm sounds, cut off hand switch.

25. Check that N-29 is ready to receive the HNO_3 rinse.

26. Jet the HNO_3 rinse from R-79 to N-29. N-29 is now ready for the volume reduction step of the Cerium Extraction Procedure.

NOTE: Section C below can be done during the volume reduction step for N-29.

C. Removal of Organic Phase

1. Check R-29 to see that all valves and jets are off and that tank is ready to receive organic ^{91}Y waste from N-19.

2. Transfer the organic waste from N-19 to R-29. Set air flow to airlift at 50%.

3. Add 35 gallons of H_2O to N-19 and agitate for 5 minutes and transfer to R-29. Set air flow to airlift at 50%.

NOTE: R-29 should have a liquid level of 40% by volume to prime U-91 organic pump. This pump should not be run dry. The interlock stops pump when liquid level reaches 5% by volume.

4. Pump the organic ^{91}Y waste from R-29 to W-6. Check with supervision.

5. Follow pumping of organic waste with 50 gallons of water wash to W-6.

6. Continue with Cerium Extraction Procedure.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERIUM-RARE EARTH SEPARATION

DATA SHEET NO. 1

(Yttrium Extraction)

Batch Number _____ Time/date started _____

Status (after feed transfer to Cell 9)

Feed batch identification _____

H89 vol. _____ l Sample code _____

Operation to follow this batch _____

_____

Operation

N19 vol. _____ l Sample code _____

Normality adjustment:

Chemical Addition	N19 Norm.	Chemical Addition	N19 Norm.
8 l 19M NaOH			

Vol. D2EHPA added _____ l Vol. Amsco added _____ l

Digestion times: started _____ Completed _____

Final volume N29 _____ l

Volume organic waste N19 _____ l

Date/time organic waste discarded to W6 _____

Remarks _____

_____

_____

_____

Time/Date completed _____

Supervisor _____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERIUM EXTRACTION

A. Chemical Makeup

1. 3 gallons of 19 M NaOH (C. P. grade).
2. 10 liters of 1.0 M $KMnO_4$ (1580 grams of $KMnO_4$ and 60 milliliters of 70% HNO_3 added to 10 liters of hot water, 65°C).
3. 16 gallons of 2 M HNO_3.
4. 33.0 liters of Amsco.
5. 9.9 liters of Amsco.
6. 16.5 liters of Amsco.
7. 6.6 liters of D2EHPA.
8. 10 liters of 1 M citric acid.
9. 30 gallons of 1 M HNO_3 (15 gallons of 2 M HNO_3 diluted with 15 gallons of water).
10. 26.4 gallons of 10 M HNO_3.
11. 3 liters of 30% H_2O_2.

B. Operation

1. Record N-29 volume with agitator off. Assume specific gravity of 1.1.
2. Turn on N-29 agitator and heat solution to 95°C.
3. Reduce N-29 volume to 90 liters, then cool to 25°C.
4. With agitator on, sample N-29 solution for the lab. Code _____Cel.
5. Turn off N-29 agitator and record the volume on batch card. Assume a specific gravity of 1.1.
6. Turn on agitator and sample N-29 solution for acid normality by titration in Cell 19.
7. NOTE: If more than 8 hours have elapsed since volume reduction at 95°C, digest N-29 at 90°C for 2 hours and cool to 25°C. If less than 8 hours have elapsed, continue with next step.

8. Adjust acid normality to 1 N ± 0.05 N free acid. Add 250 milliliters of acid or base for each 0.05 change in normality. Rinse addition line with 2 liters of H_2O. Hold temperature at 20 to 25°C with water on jacket.

9. Add 1 M HNO_3 to bring volume up to 100 liters.

10. Turn off agitator and record volume of aqueous solution in N-29 on Batch Data Sheet. Assume a specific gravity of 1.1.

11. Make up 10 liters of 1 M $KMnO_4$ by adding 1580 grams of $KMnO_4$ to 10 liters of hot water. Acidify $KMnO_4$ solution with 60 milliliters of 70% HNO_3.

    NOTE: Operations have to proceed rapidly from step to step from here on.

12. Turn agitator on and jacket water on N-29.

13. Add 5 liters of 1 M $KMnO_4$ to N-29.

14. Add 5 liters of 2.0 M HNO_3 solution to N-29.

15. Add 6.6 liters of D2EHPA to N-29.

16. Add 9.9 liters of Amsco to N-29.

17. Agitate for 15 minutes from time of last Amsco addition.

18. NOTE: Sample N-29 every 5 minutes before going to Step 19. Place several drops of aqueous solution (no organic) on a piece of filter paper. The manganese dioxide (brown color) will stay where the drops of solution hit the filter paper. The solution will soak outward from this spot on the filter paper. If there is an excess of $KMnO_4$, a light pink color will be observed. Add 1.0 liter of 1 M $KMnO_4$ at a time as needed to keep a slight pink color. Record on Batch Data Sheet the total volume of extra 1 M $KMnO_4$ added. Wash each liter of $KMnO_4$ with 1.0 liter of 2 M HNO_3.

19. At end of digestion period, immediately add 16.5 liters of Amsco and agitate for 15 minutes. Also see Note 18 in regard to maintaining color.

20. At end of digestion period, immediately add 5 liters of 1 M citric acid to N-29 (2100 grams of citric acid in 10 liters H_2O.

21. NOTE: If extra $KMnO_4$ was used, add an extra liter of 1 M citric acid for each extra liter of 1 M $KMnO_4$ used to keep pink color.

22. Agitate for 10 minutes after addition of citric acid.

23. Turn off agitator and let N-29 settle for 10 minutes.

24. Check R-79 that all jets are off and tank is ready to receive solution from N-29.

25. Open valve from N-29 to R-79 and separate aqueous phase from the organic by airlift flow of aqueous to R-79. Set air flow to airlift at 37.5% to give a flow of about 3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and the valve from N-29 to R-79 will automatically close.

26. When the alarm sounds, cut off hand switch.

27. Check N-19 that all valves and jets are closed and tank is empty and ready to receive solution from R-79.

28. Jet the aqueous solution containing promethium from R-79 to N-19 and hold.

29. Add 1.5 gallons of 1.0 M HNO_3 to N-29 and let settle for 3 minutes.

30. Open valve from N-29 to R-79 and separate aqueous phase from the organic by airlift flow of aqueous to R-79. Set air flow to airlift at 37.5% to give a flow of 3.0 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and the valve from N-29 to R-79 will automatically close.

31. When the alarm sounds, cut off hand switch.

32. Jet the aqueous solution from R-79 to N-19 and hold.

33. Add 1.5 gallons of 1.0 M HNO_3 to N-29 and let settle for 3 minutes.

34. Open valve from N-29 to R-79 and separate aqueous phase from the organic by airlift flow of aqueous to R-79. Set air flow to airlift at 37.5% to give a flow of 3.0 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and the valve from N-29 to R-79 will automatically close.

35. When the alarm sounds, cut off hand switch.

36. Jet the aqueous solution from R-79 to N-19 and hold for the Silica Manganese and Nickel Removal Procedure.

C. Acid Rinse of Organic Phase

1. Add 26.5 gallons of 10 M HNO_3 to N-29.

2. Agitate solution in N-29 for 10 minutes at 25°C.

3. Cut off agitator and let N-29 settle for 10 minutes.

4. Check R-79 that all jets are off and tank is ready to receive solution from N-29.

5. Open valve from N-29 to R-79 and separate aqueous phase from organic. Set air flow to airlift at 37.5% to give a flow of ~3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface instrument, the alarm will sound and the valve from N-29 to R-79 will automatically close.

6. When alarms sound, close hand switch.

7. Check R-39 that all jets and valves are closed and tank is empty.

8. Jet acid rinse from R-79 to R-39.

9. Add 2.5 gallons of 1 M HNO_3 to N-29.

10. Open valve from N-29 to R-79 and separate aqueous phase from organic. Set air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface instrument, the alarm will sound and the valve from N-29 to R-79 will automatically close.

11. When the alarm sounds, cut off the hand switch.

12. Jet the acid rinse from R-79 to R-39.

13. Add 2.5 gallons of 1.0 M HNO_3 to N-29.

14. Open valve from N-29 to R-79 and separate aqueous phase from organic. Set air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic phase reaches the short leg on the interface instrument, the alarm will sound and the valve from N-29 to R-79 will automatically close.

15. When the alarm sounds, cut off the hand switch.

16. Jet the acid rinse from R-79 to R-39 and agitate.

17. Sample R-39 for the lab. Code_____CelW.

18. Cut off R-39 agitator and then record R-39 volume on the Batch Data Sheet and assume a specific gravity of 1.1.

19. Jet R-39 solution to W-6.

D. Re-Extraction of ^{144}Ce from Organic Phase

1. Add 33.0 liters of Amsco to N-29.

2. Add 8 gallons of 2 M HNO_3 to N-29. Immediately add 3 liters of H_2O_2.

3. Agitate N-29 for 10 minutes at 25°C.

4. Cut off agitator and let N-29 settle for 10 minutes.

5. Check R-79 that all jets are off and that tank is empty and ready to receive the ^{144}Ce fraction.

6. Open valve from N-29 to R-79 and separate the aqueous from organic by airlift to R-79. Set air flow to airlift at 37.5% to give a flow of ~3 gallons/minute.

NOTE: When the organic reaches the interface probe, the alarm will sound and the valve from N-29 to R-79 will close automatically.

7. When alarm sounds, close hand switch.

8. Check H-511 in Cell 11 that all valves and jets are closed.

9. Check that H-511 is empty and ready to receive the ^{144}Ce solution from R-79.

10. Jet R-79 to H-511.

11. Add 1.5 gallons of 1 M HNO_3 to N-29.

12. Open valve from N-29 to R-79 and separate the aqueous from organic by airlift to R-79. Set air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic reaches the interface probe, the alarm will sound and the valve from N-29 to R-79 will close automatically.

13. When the alarm sounds, close the hand switch.

14. Jet R-79 to H-511.

15. Add 1.5 gallons of 1 M HNO_3 to N-29.

16. Open valve from N-29 to R-79 and separate the aqueous from organic by air lift to R-79. Set air flow to airlift at 37.5% to give a flow of 3 gallons/minute.

NOTE: When the organic reaches the interface probe, the alarm will sound and the valve from N-29 to R-79 will close automatically.

17. When the alarm sounds, close the hand switch.

18. Jet R-79 to H-511, which is now ready for the Cerium Precipitation Procedure.

19. Open valve from N-29 to R-29 and transfer to organic waste to R-29 by setting the air flow to airlift at 50%.

20. Add 36 gallons of H_2O to N-29 and agitate for 5 minutes.

21. Transfer solution from N-29 to R-29.

NOTE: R-29 should have a liquid level of 40% by volume to prime U-91 organic pump. This pump should not be run dry. The interlock will stop the pump when liquid level reaches 5% by volume.

22. Cut off starter switch.

23. Pump the organic waste from R-29 to W-6. Check with supervision.

24. Follow pumping of the organic waste with a 50-gallon water wash.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERIUM-RARE EARTH SEPARATION

DATA SHEET NO. 2

(Cerium Extraction)

Batch Number_____ Time/date started_____

Status (before starting operations)

Feed batch identification_____

N29 volume_____ l Operation to follow this batch_____

Operation

Evaporation

Time started_____ Time completed_____

N29 vol._____ l (after evap.) Sample code_____

^{144}Ce Extraction

Normality adjustment:

Added:	N29 Norm.	Added:	N29 Norm.	Added:	N29 Norm.

Vol. 1.0 N HNO_3 added_____ l Adjusted vol. N29_____ l

Chemical additions:

_____ l 1M $KMnO_4$ (1st add.), _____ l 2N HNO_3, _____ l D2EHPA

_____ l Amsco (1st add.), _____ l 1M $KMnO_4$ (during digest)

_____ l Amsco (2nd add.), _____ l citric acid

Digestion times: Started_____ Completed_____

Phase separation and rinse times: Started_____ Completed_____

Vol. aqueous phase in N19_____ l

Acid rinse:

Rinse solution	Vol.	Time added to N29	Time transferred to R79

Vol. acid rinse in R-39_____ l Sample code_____

Time/date acid rinse discarded to W6_____

Re-extract of ^{144}Ce

Chemical additions: (time started_____)

_____ l Amsco, _____ l 2N HNO_3, _____ l H_2O_2

Phase separation and acid rinse times: Started_____ Completed_____

Final volume H511_____ l

Time/date organic waste discarded to W6_____

Remarks_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERIUM PRECIPITATION

NOTE: The starting point for this operation is after the ^{144}Ce feed has been transferred from Cell 9 to H511.

A. Chemical Makeup

1. 6 liters of concentrated NH_4OH.

NOTE: Oxalic acid should be made in advance.

2. 10 liters of 1.8 M oxalic acid. (For makeup, weigh out 5 pounds of oxalic acid and add 10 liters of hot water. Agitate until oxalic acid is in solution.)

B. Solution Adjustment

1. Check Cell 9 procedure on re-extraction of ^{144}Ce from the organic phase and ^{144}Ce transfer to Cell 11.

2. Do not exceed three batches of ^{144}Ce in Cell 11 at one time.

3. Check that P511 is empty and all valves and services are closed.

4. Assemble filter for P511 according to Ceramic Filter Buildup Procedure.

5. Jet one batch of ^{144}Ce product from H511 to P511 and turn on P511 agitator.

NOTE: One batch of ^{144}Ce product from Cell 9 should amount to approximately 13.5 gallons and should contain approximately 75 moles of free HNO_3 acid, with jet dilution.

6. Add 1 gallon of water to H511 and jet to P511 if H511 has only one batch.

7. Repeat step 6.

8. Record P511 volume with agitator off.

9. Turn on P511 agitator.

10. Sample P511 in Cell 19 after 10 minutes of agitation. Sample Code_____OX3. (Dilute 1 to 500 milliliters.)

11. Take 5 milliliters of original sample and titrate for acid normality with 1.0 M NaOH.

12. Heat P511 to 50°C with agitation. Set steam regulator valve to P511 jacket on panelboard and open steam outlet and inlet valves located on cell face.

13. Using schedule listed below, adjust acid normality to 0.2 N ± 0.1 N free acid with concentrated ammonium hydroxide (NH_4OH), while holding temperature at 50°C and off-gas between 6 and 8 inches.

    First addition: 3 liters slowly - ~5 minutes.
    Second addition: 1 liter slowly - ~5 minutes.
    Third addition: 0.2 liter slowly- ~2 minutes.

NOTE: The above additions of NH_4OH were calculated on a volume of 60 liters or 15.85 gallons of starting solution in P511 and concentrated NH_4OH at 14 M.

C. Oxalic Acid Precipitation of ^{144}Ce

1. Heat solution in P511 to 80°C with agitation. Set regulator on panelboard to 80°C and open steam inlet and outlet valves located on cell face.

2. Be sure that oxalic acid is in solution and that there is no delay in the procedure once the oxalic addition to P511 has been started.

3. At 80°C add 2 liters of 1.8 M oxalic acid to P511.

4. Cool P511 to 60°C over a period of 15 minutes. Set steam controller to 0 on panelboard and close manual valves on cell face. Open water outlet valve from jacket. The water inlet valve should be open for 1-minute intervals to prevent cooling P511 too fast.

5. At 60°C add 4 liters of 1.8 M oxalic acid to P511.

6. Cool P511 to 40°C over a period of 15 minutes.

7. At 40°C add 4 liters of 1.8 M oxalic acid to P511 and hold at 40°C for 10 minutes.

8. Cool P511 to 25°C and leave water on until after filtration.

9. Check step D.1 to estimate the time for beginning the filtration.

10. Begin adding 1 liter of 1.8 M oxalic acid every 30 minutes.

11. If filtration is to be delayed for as much as 2 hours, notify supervision.

D. Filter Operation

1. Check R111 that all valves and services are closed except off-gas and that vessel is empty.

2. Turn on the steam jet and close off-gas valve.

3. Open the P511 bottom discharge valve and begin filtration.

4. When P511 liquid level reaches 10% of tank capacity, turn off agitator.

NOTE: Vacuum on R111 should decrease when all solution has been filtered from P511 to R111.

5. After the filtration has been finished, rinse P511 with water spray, two 1-minute portions.

6. Continue vacuum jet until a vacuum break on R111 is noted for each water rinse.

7. After water rinse, close P511 bottom discharge and turn off water to P511 jacket.

8. Turn off R111 vacuum jet and open R111 off-gas.

9. Sample R111 Code_____0X3W. Record volume.

10. Hold solution in R111 for sample results. Supervision will determine disposition of solution.

E. Calcination

1. Disassemble the filter. Remove the ceramic filter thimble.

2. Allow the precipitate to dry. When the precipitate is dry, as indicated by no more steam coming off it, stir the powder and scrape down the powder from the sides of the filter.

3. Allow the precipitate to stand for at least 1 hour after steam has stopped. Stir the powder once every 15 minutes.

4. Transfer the ceramic filter to the furnace and close the furnace door.

5. Set the 700°C template on the furnace controller.

6. Turn on the power to the furnace and turn on the controller.

NOTE: The furnace will heat at a rate of 100°C per hour to 700°C and maintain the 700°C temperature for 2 hours. Record data on furnace record sheet.

7. When the furnace has cooled to <300°C, open the furnace door.

8. Check with supervision for instructions on powder weighing, assay, and storage.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERIUM PRECIPITATION DATA

DATA SHEET

Batch Number_____ Time/date started_____

Status (before starting operation)

Feed batch identification_____

H511 volume_____liters Sample code_____

Operation

P511 volume before normality adjustment_____liters

Final normality P511 soln._____ N: Vol. P511_____liters

P511 volume after oxalic addition_____liters

Filtration times: Started_____ Completed_____

Filtrate (R111) volume_____liters: Sample code_____

Calcination times: Started_____ Completed_____

Product Data

Total weight product_____grams

Calorimeter results: 1st check_____c/g 2nd check_____c/g

Total ^{144}Ce curies_____ Total ^{144}Ce watts_____

Product disposition_____

Remarks_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

SILICA, MANGANESE, AND NICKEL REMOVAL

A. Chemical Makeup

1. 4 gallons of 19 M NaOH (C.P. grade).
2. 19.8 liters of Amsco.
3. 13.2 liters of D2EHPA.
4. Dilute acid rinse (2 milliliters of 16 M HNO_3 to 4 liters of water).
5. 133 liters of 2 M HNO_3.
6. 33 liters of Amsco.

B. Promethium Extraction of Feed

1. Reduce volume in N19 to 80 liters by heating to 95°C.
2. When volume in N19 is 80 liters, cool to 25°C.
3. Add 4.0 liters of 19.0 M C.P. NaOH to N19 and follow with a 2-liter water rinse.
4. Titrate sample for acid normality.
5. Adjust acid normality to 0.1 N by adding 500 milliliters of 19 M NaOH or 500 milliliters of 70% HNO_3 for each 0.1 N to be raised or lowered, and follow each addition with a 2.0-liter water wash.
6. Adjust pH to 1.6 to 2.3 by adding 125-milliliter increments of 19.0 M C.P. NaOH; follow each addition with a 2.0-liter water rinse.
7. Record final pH on the Batch Data Sheet.
8. Add 13.2 liters of D2EHPA.
9. Add 19.8 liters of Amsco.
10. Agitate for 10 minutes.
11. After a 9-minute agitation period, jet sample to Cell 19 for examination. If phases do not separate satisfactorily in 10 minutes in the sample tube, check with supervision.
12. Turn off agitator and let N19 settle for 10 minutes.

13. Take sample in Cell 19.

14. The organic liquid may cause pH meter trouble. Attempt to separate at least most of the organic liquid from the aqueous before checking the pH of the aqueous liquid.

15. Check pH of solution.

16. Adjust pH to 1.6 to 2.3 by adding 125-milliliter increments of 19 M C.P. NaOH; follow each with a 2.0-liter water rinse.

17. Agitate N19 for 10 minutes.

18. Let N19 settle for 10 minutes.

19. Take sample in Cell 19.

20. The organic liquid may cause pH meter trouble. Attempt to separate at least most of the organic liquid from the aqueous before checking the pH of the aqueous liquid.

21. Check pH of solution.

22. Adjust pH to 1.6 to 2.3 by adding 125-milliliter increments of 19 M C.P. NaOH until the pH remains at 1.6 to 2.3. Follow each NaOH addition with 2.0-liter water rinse.

23. When pH is in range of 1.6 to 2.3, continue with procedure.

24. Check R-79 to be sure that all jets are off and tank is ready to receive solution from N19.

25. Proceed only after N19 has been allowed to settle for 10 minutes after the last sample has been taken.

26. Open valve from N19 to R79 and separate aqueous phase from the organic by airlift flow of aqueous to R79. Set air flow at 37.5% to give a flow of about 3 gallons/minute to airlift.

27. When the organic phase reaches the short leg on the interface, the alarm will sound and valve from N19 to R79 will close automatically.

    When the alarm sounds, cut off hand switch.

28. Jet solution from R79 to R39.

NOTE: At this time the feed material in R39 has the promethium product removed from it and the organic solution contains the promethium product.

29. Sample solution in R39 for the lab. Code_____SilW.

30. Jet R39 solution to W6.

31. Add 50 liters of 2 M nitric acid to N19.

32. Add 33 liters of Amsco to N19.

33. Agitate N19 for 15 minutes.

34. Turn off agitator and let N19 settle for 10 minutes.

35. Check R79 that all jets are off and tank is ready for solution from N19.

36. Open valve from N19 to R79 and separate aqueous phase from the organic by airlift flow of aqueous to R79. Set air flow at 37.5% to give a flow of about 3 gallons/minute to air lift.

37. When the organic phase reaches the short leg on the interface, the alarm will sound and valve from N19 to R79 will close automatically.

    When alarm sounds, cut off hand switch.

38. Check that all valves to F2 and P89 are closed and P89 has off-gas.

39. Pressurize the bottom of F2 filter with air regulator at 5 psi with valve 10933 open.

40. Jet R79 to P89.

41. Add 50 liters of 2 M nitric acid to N19.

42. Add 33 liters of Amsco to N19.

43. Agitate N19 for 15 minutes.

44. Turn off agitator and let N19 settle for 10 minutes.

45. Check R79 that all jets are off and tank is ready for solution from N19.

46. Open valve from N19 to R79 and separate aqueous phase from the organic by air flow of aqueous to R79. Set air flow at 37.5% to give a flow of about 3 gallons/minute to airlift.

47. When the organic phase reaches short leg on interface, the alarm will sound and valve from N19 to R79 will close automatically.

    When the alarm sounds, cut off hand switch.

48. Jet R79 solution to P89, which is now ready for the Promethium Oxalate Procedure.

C. Organic Removal Procedure

1. Open valve from N19 to R29 and transfer the organic waste to R29 by regulating the air flow to 50% on the airlift.

2. Add 36 gallons of water to N19 and agitate for 5 minutes and then transfer to R29.

   R29 should have a liquid level of 40% by volume to prime U91 organic pump. This pump should not run dry. The interlock will stop pump when liquid level reaches 5% by volume.

   Cut off starter switch.

3. Pump the organic waste from R29 to W6. Check with supervision.

4. Follow organic with 50-gallon water wash.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CERIUM-RARE EARTH SEPARATION

DATA SHEET NO. 3

(Si, Mn, Ni Removal)

Batch Number_____ Time/date started_____

Status (before starting operations)

Feed batch identification_____

N19 vol._____ Sample code_____

Operations to follow this batch_____

_____

Evaporation

Operation

Evaporation times: Started_____ Completed_____

N19 Vol._____ l (after evap.) Sample code_____

^{147}Pm Extraction

Normality adjustment:

Added:	N19 Norm.	Added:	N19 Norm.	Added:	N19 Norm.
4 l 19M NaOH					

Vol. D2EHPA added_____ l, Vol. Amsco added_____ l

Digestion/settling times:

Digest No.	Time started	Agitator off at:	Time sampled	Total chemicals added for pH adjust.
1				
2				

Phase separation and rinse times: Started_____ Completed_____

R39 vol._____ l Sample code_____ Time/date discarded to W6_____

Re-extraction of ^{147}Pm

Vol. 2 N HNO_3 added_____ l, Vol. Amsco added_____ l

1st digestion times: Started_____ Completed_____

Volume aqueous in P89_____ l, (after 1st re-extraction)

Vol. 2 N HNO_3 added_____ l, Vol. Amsco added_____ l

2nd digestion times: Started_____ Completed_____

Volume aqueous in P89_____ l, (after 2nd re-extraction)

Time/date organic waste discarded to W6_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

PROMETHIUM PRECIPITATION

A. Chemical Makeup

1. 3.0 gallons of 19 M NaOH (C.P. grade).
2. 4 liters of 1.8 M oxalic acid (1/2 pound/liter).
3. 4 liters of 7 M HNO_3.
4. 3 milliliters of 80% $Mn(NO_3)_2 \cdot 6H_2O$.

B. Operation

1. Agitate solution in P89 for 5 minutes and record volume with agitator off.
2. Sample P89. Code_____OX2.
3. Turn on P89 agitator and adjust the solution to 0.25 to 0.30 N free acid by adding 500 milliliters of 70% HNO_3 or 500 milliliters of 19 M C.P. NaOH for each 0.1 N to be raised or lowered while holding the temperature at 20 to 30°C and the off-gas between 6 and 8 inches.
4. Heat P89 to 80°C.
5. Make up 4 liters of 1.8 M oxalic acid (2 pounds of oxalic acid in 4 liters of hot water, 80°C).
6. Add 2 liters of above oxalic acid to P89. Follow oxalic acid with 2 liters of water.
7. Cool P89 solution 10°C every 10 minutes until temperature reaches 40°C. When solution reaches 40°C, add 2 liters of oxalic acid. Then continue cooling and agitation for 1.5 hours. Test for complete precipitation as outlined in steps 7.a to 7.c below:

   a. Take a 10-millimeter sample and centrifuge. Pour the supernate into a clean centrifuge tube. Add 1 milliliter of saturated oxalic acid solution and stir. Centrifuge. If no precipitate is formed, the rare-earth oxalate precipitation is complete.

   b. If a white precipitate forms, add 10 milliliters of water to precipitate and stir to dissolve. Centrifuge. If this precipitate dissolves, it is sodium oxalate.

   c. Only the promethium oxalate appears green in the cell and is an indication of an incomplete precipitation. If the precipitation is not complete, make up 2 liters of 1.8 M oxalic acid (1 pound of oxalic acid in 2 liters of hot water). Add to P89 and digest for 1 hour and then repeat test.

8. Cut off air pressure to F2 by closing valve 10933, and set air regulator at 5 psi.

9. Check that R39 is empty and ready to receive the filtrate from F2.

10. Start vacuum jet and open valve from F2 to vacuum jet. Open valve from P89 to F2.

11. Filter out the precipitate in F2.

12. Cut off P89 agitator when liquid level reading is approximately 20% by volume.

13. Record filtration rate.

14. Observe volume of R39 during filtration. The jet dilution may overfill tank.

15. Continue vacuum jet according to instructions from supervision. This will be determined by filtration rate. F2 has approximately 2 3/4 gallons holdup capacity.

16. After filtration is finished, rinse P89 with water spray, two 2-minute portions.

NOTE: Flow rate on P89 water spray is approximately 0.8 gallons/minute.

17. Continue vacuum jet for calculated time on each of the water rinses from P89.

18. It is essential that no liquid remain in F2 before going to step 19. Let vacuum jet run 15 minutes or 20% of total filtration time, whichever is the <u>longer</u> after F2 should be dry.

19. Cut off vacuum jet to R39 and close valve from F2 to vacuum jet. Also close valve from P89 to F2.

20. Record R29 volume and sample filtrate in R29. Code_____OX2W.

21. Jet R39 solution to waste.

22. Add 50 gallons of water to R39; then jet R39 to waste.

C. <u>Precipitate Dissolving in F2</u>

1. Check all valves to and from F2 to see that they are closed.

2. Open off-gas valve from F2 to P89, off-gas condenser water, and P89 jacket water.

3. Do not close off-gas valve during dissolving in F2.

4. Pressurize the bottom of F2 filter to 8 inches of H_2O by opening valve on air line and pressure regulator valve to 0.3 pound on panelboard.

NOTE: Air pressure is to be maintained on the bottom side of F2 during dissolving of precipitate. This is to prevent acid from leaking through filter.

5. Add 5 gallons of 70% HNO_3 and 15 milliliters of 80% $Mn(NO_3)_2 \cdot 6H_2O$ to P89. Open valve from P89 to F2 to drain acid solution to F2.

6. When sampling F2, leave jet open only a minimum time to get sample.

7. Leave valve open from P89 to F2 after acid has drained to F2.

8. Heat F2 to 95°C and digest for 4 hours.

9. Cool F2 to 25°C.

10. If requested, sample F2. Solution Code_____PM1.

11. Record F2 volume on Batch Data Sheet. This volume can be obtained by reading P89 liquid level and getting the volume from F2 calibration.

D. Product Transfer Procedure to Cell 15

NOTE: All cell entry procedures apply. Check with supervision.

1. Cap-off long leg of product carrier.

2. Make up a 00 rubber stopper with a hole in bottom to prevent valve from opening when placed in Snaptite on long leg of product carrier.

3. Place promethium carrier on 10-ton dolly in Cell 15.

4. Connect Snaptite fitting on short leg of carrier from Cell 9.

5. Connect Snaptite fitting on short leg of carrier from Cell 15 to P45, P12, E16 (jet No. J56-1).

6. Check that P12 is empty and will not be in operation for ~3 to 4 hours.

7. Open valve in Cell 15 on suction side of jet J56-1.

8. Open valve on discharge of J56-1 to P12.

9. Close valve on air line to bottom of F2 and set pressure regulator to 0.

10. Open valve from F2 to Cell 15 on Panelboard 4.

11. Open manipulator-operated valve in Cell 15 from F2.

12. Turn on jet J56-1.

13. Time for transfer may vary; so check F2 by sampling after product has been transferred to be sure filter vessel is empty.

14. Rinse F2 with two 4-liter portions of 70% C.P. HNO_3. Transfer each rinse to product carrier.

15. After rinses, disconnect line to vacuum jet J56-1 and flush with water to P12.

16. Close valve to Jet J56-1 and manipulator-operated valve in Cell 15.

17. Cut off steam jet No. J56-1 and close valve to P12.

18. Close panelboard valve F2 to Cell 15.

19. Turn off F2 off-gas condenser water.

20. If the filtration through F2 was slow, check with supervision for a filter cleanup procedure.

NOTE: After each transfer the Tygon lines are to be discarded but the Snaptites are to be decontaminated and new 6-feet-long Tygon lines put on.

FISSION PRODUCTS DEVELOPMENT LABORATORY

PROMETHIUM PRECIPITATION

DATA SHEET

Batch Number_____ Time/date started_____

Status (before starting operation)

Feed batch identification_____

P89 vol._____ l Sample code_____

Operation

Precipitation

Normality adjustment:

Added:	P89 Norm.	Added:	P89 Norm.	Added:	P89 Norm.

Vol 1.8 M oxalic acid added_____ l

Digestion times: (start as of start of cooling operation)

Temp.	Time	Temp.	Time	
80°		50°		
70°		40°		(begin digest.)
60°		40°		(end digest.)

Filtration times: Started_____ Completed_____

Vol R39 after filtration and rinse_____ l Sample code_____

Time/date waste discarded to W6_____

Precipitate Dissolving

Vol 16 N HNO_3 added_____ l, Vol $Mn(NO_3)_2$ added_____ ml

Digestion times: Started_____ Completed_____

Volume F2_____ l Sample code_____

Time/date product transferred to carrier_____

Remarks_____

Supervisor_____

FISSION PRODUCTS DEVELOPMENT LABORATORY

SAFETY REGULATIONS

A. Protective Clothing and Equipment

1. General building safety requirements are safety glasses, safety shoes, film badges, and pocket meters.

2. Chemical handling and mixing require coveralls with the sleeves fastened at the wrist, face shields or respirators (for dry chemicals), and rubber gloves. When handling nitric acid and sodium hydroxide, rain suits and hat are also required.

3. Hard hats will be worn whenever it is necessary to enter a cell or pipe tunnel when the roof blocks are removed, and in any construction area.

B. Handling and Storing Chemicals and Equipment

1. Fifty pounds is the limit that can be handled by one man. Items weighing over 50 pounds will be handled by two or more men or by the crane or other material-handling equipment.

2. Loading areas will be chained off when equipment or chemicals are being lifted with the crane.

3. Gas cylinders will be handled according to standard procedure, which includes keeping the cylinders secured in upright position and keeping the valves capped. All empties will be tagged and placed in the "empty" racks.

4. Carboys will be handled by carboy lifters and drum holders and will be flushed and tagged after being emptied.

5. Acid bottles will be carried in approved containers, and the bottles will be cleaned immediately after they are emptied.

6. All empty chemical containers will be removed from the building and stored in proper places as soon as possible.

7. Hydrazine will be less than 42.5% concentration and will be handled and stored as described in the "Chemical Makeup" procedure.

8. Solvents will be stored as follows:

   a. All solvents will be stored outside under the solvent storage shed, and no solvents will be stored inside the building.

b. Lubricating oils will be stored in the cabinet provided, and any excess should be stored under the solvent storage shed.

c. No flammable material will be stored on stairway storage areas.

C. Spills and Leaks

1. Spills and leaks of any kind are to be investigated, monitored, and cleaned up at once.

2. Every effort should be made to avoid leaks and spills that will contact electric switches and equipment and cause short circuits.

3. Every spill should be considered a possible radiation and contamination hazard and handled accordingly.

D. Mechanical and Electrical Equipment

1. Electrical lockout:

a. Main electrical switches will be turned off, locked, and tagged whenever work is necessary on electrical equipment. Electrical permits will be signed and followed as directed.

b. Danger tags will not be removed from equipment by anyone except the person who originally placed them on the equipment unless that person or higher supervision is first contacted for approval.

c. No power-operated equipment will be operated until the operator personally clears the area of personnel. This includes cranes, pumps, centrifuges, agitators, and other such equipment.

2. Crane operation:

a. The crane operator will ascertain that adequate slings are used to lift the load, that the load will not swing when lifted, and that the load will not strike any object during its travel. In addition, he must ascertain that the load will be set on adequate support, keeping in mind that Cells 14 and 15 have limited supporting beams.

b. When not in use, the bridge crane will always be parked over the loading bay with the crane hook at least 10 feet above the floor.

c. When the power is on the crane, care must be taken to avoid contact between remote tool rods and the crane rail. The crane power should be shut off when remote tool rods are being used.

3. Miscellaneous:

   a. When lifting cell blocks, adequate slings should be used and the lifting eyes should be screwed tightly in place.

   b. Blocks are not to be set over Cell 14 or Cell 15.

   c. STT's and HAPO's should be moved very carefully in accordance with procedures.

   d. All broken and contaminated ladders should be bagged and sent to the burial ground. Excess ladders should be removed from the building. Aluminum ladders should be considered an electrical hazard and handled accordingly.

E. Fire Fighting and Emergency Equipment

1. Evacuation procedure as posted will be followed.

2. Access to safety showers and emergency equipment will always be maintained.

3. All personnel will acquaint themselves with the location and operation of fire fighting equipment, emergency equipment, safety showers, and assault and air-line masks.

FISSION PRODUCTS DEVELOPMENT LABORATORY

EMERGENCY AND EVACUATION PROCEDURE

A. Building Evacuation Systems

1. Fire or General Emergency Evacuation System

The building evacuation alarm signal is a siren located in the crane bay. It is activated by either one of two evacuation switches; one is located on the central alarm panel in the shift supervisor's office and the other is on the south wall of the manipulator operating gallery.

2. Contamination Alarm Evacuation System

The building contamination alarm signal is a high-pitched air whistle (clarion whistle) located in the crane bay. It is activated automatically when any two of the four constant-air monitors in the building detect a high level of air contamination. This signal can also be activated manually by a switch located on the contamination alarm panel in the shift supervisor's office.

B. Emergency Procedure (For All Personnel)

1. Any employee discovering a fire or explosion will proceed as follows:

   a. Activate the nearest fire alarm box and/or call the ORNL emergency number (3-6358) and describe the circumstances.

   b. Notify or have someone notify the Local Emergency Supervisor.

   c. Attempt to control the fire if it is practical to do so.

   d. Meet or have someone meet the firefighters and direct them to the scene of the fire.

   e. Cause the evacuation alarm to be sounded if it is obvious that the building should be evacuated.

   NOTE: A small fire (one which the employee is completely sure that he can extinguish) may be extinguished without adherence to the above. The employee will then notify the Local Emergency Supervisor, who will contact the Fire Department.

2. Any employee discovering an emergency situation other than fire or explosion will proceed as follows:

Immediately cause the local evacuation signal to be sounded. All operators will observe the following procedure:

a. Stop work and shut off power in own work area. If it is not practical to shut off power, shut off any electrical machines and turn off lights.

b. Close all doors in own work area except those to be used as exits or opening upon or under exits.

c. Rapidly walk out of the building, following the posted evacuation route if possible. If the posted evacuation route is blocked, leave the building by the shortest safe route.

NOTE: While leaving the building, personnel should take any portable radiation instruments that are in the area with them.

d. Proceed to the assembly point on White Oak Avenue, southwest of the FPDL, unless assigned to local Combat Squad duties.

e. From the assembly point, proceed to evacuation routes as instructed by the Local Emergency Supervisor, or follow any instructions given over the public address system.

2. Upon hearing either of the building evacuation signals or an announcement of local evacuation on the building public address system, nonemergency personnel will proceed as described in Steps C.1.a through C.1.e above. Local Combat Squad members and searchers will assemble at the FPDL emergency control area in the southwest corner of the west airlock.

3. During any evacuation, all employees are to abide by the following rules:

a. Don't run.

b. Don't crowd the person ahead.

c. Don't lag behind and thus slow or break the column.

d. Don't scream, laugh, or talk, causing unnecessary noise.

e. Don't cause confusion by becoming unnecessarily excited.

f. Don't remain in toilet or dressing room.

g. Don't return for clothing or personal items.

h. Don't attempt to leave assembly point until dismissed or until it is permissible to return to the building.

i. Don't attempt to leave building except in accordance with evacuation drill regulations.

j. Don't fail to follow instructions.

D. Duties of Building Emergency Personnel

1. Local Emergency Supervisor

The Local Emergency Supervisor for the FPDL area is the FPDL Operations Supervisor. During emergency situations he will have direct charge of the area and will directly combat emergencies until relieved by the Laboratory Shift Supervisor. Specific duties of the Local Emergency Supervisor are as follows:

a. He will coordinate and supervise the training of FPDL personnel, both combat squads and nonemergency personnel, in matters relating to evacuation and emergency situations.

b. He will have general charge of all matters pertaining to evacuation drills and organization.

c. He will prepare and have conspicuously posted in each area of the building a notice of the evacuation route for that area.

d. He will periodically inspect the area to ensure that exit facilities conform to the requirements of the Safety and Fire Departments.

e. He will be responsible for planning emergency shutdown measures for processes and equipment.

f. He will assign specific personnel as area combat squad members and as searchers and will post these assignments.

g. He will establish requirements for the type, amount, and location of emergency equipment and supplies, and will ensure that such materials are available at all times.

2. Alternate Emergency Supervisor

The Alternate Emergency Supervisor is the FPDL Shift Supervisor. In the absence of the Local Emergency Supervisor, the Alternate Emergency Supervisor will assume all of the duties listed in D.1 above. In addition, the Alternate Emergency Supervisor will conduct training of emergency personnel on his shift.

3. Searchers

a. Searchers will be designated from each operating crew. Each searcher will be assigned a specific area.

b. During evacuation or emergency situations, searchers will report to the emergency control area in the west airlock, secure emergency equipment, and proceed to search their assigned areas.

c. Searchers will check all rooms within their areas to be sure that evacuation is complete and that all doors are closed. They will also report any unusual circumstances to the Emergency Supervisor.

d. In the event that assigned searchers are not on duty, the Local Emergency Supervisor will assign alternates.

4. Combat Squad Members

a. Combat Squad Members will be designated for each operating crew.

b. Combat Squad Members will receive specific emergency training, including the use of local firefighting equipment and other emergency equipment.

c. During evacuation or emergency situations, all Combat Squad Members will report to the emergency control area in the west airlock and secure emergency equipment. They will then combat the emergency according to instructions issued by the Emergency Supervisor.

FISSION PRODUCTS DEVELOPMENT LABORATORY

ZONING PROCEDURES

I. Regulated Zone

A. Definition

A regulated zone is an area where operations are restricted for the purpose of radioactive contamination control. This zone may contain radiation zones, contamination zones, or both, ranging in size from a small spot to a large area.

B. The FPDL Regulated Zone

The FPDL and its immediate environs consitute a regulated zone. This zone is defined as the area outlined by an imaginary straight line running north from White Oak Avenue through the east side of the FPDL cell ventilation house to the west edge of 4th Street. The northern boundary is a straight line running east and west from the north edge of the alleyway north of Building 3505 from 3rd Street to 4th Street. The zone is bounded on the west by the east edge of 3rd Street and on the north by the north edge of White Oak Avenue.

C. General Regulations

The FPDL regulated zone is accessible to all authorized personnel. Approved clothing includes personal clothing, noncontamination work clothing, and "CONTAMINATION" clothing if it has been proved free from contamination by approved checks. Entry to the regulated zone from other clean areas is unrestricted; entry from contamination zones is governed by the exit requirements of those zones. When other zones are established within the regulated zone, the regulations pertaining to these zones supersedes those of the regulated zone.

D. Control Measures

Exit from the FPDL regulated zone requires a personal contamination check at one of the monitoring stations. These monitoring stations are located at the eight personnel exits on the first floor.

Supervision must be notified when the exit check indicates personal contamination.

II. Contamination Zones

A. Definition

A contamination zone is an area where control measures are established to prevent the contamination of employees, of environs, and/or of equipment and where it is possible that radioactive material may become deposited inside the body and lead to internal radiation exposure.

B. FPDL Permanent Contamination Zones

1. All process cells.

2. All manipulator cells.

3. All tank farm cells.

4. Pipe tunnel.

5. Service tunnels (main service tunnel, Cell 20, 17, 10, service tunnel, service cubicles for Cells 11, 13, and 14).

6. Cell 20 filter cubicle.

7. Cell ventilation filter pit and house.

8. Cubicle "D" (behind barricade) - 1st level north.

9. All sample stations (Cell 18, 19, 20, tank farm sampler).

10. Tops of Cells 1 and 15.

C. FPDL Temporary Contamination Zones

Any area may be designated as a contamination zone if it is found to be contaminated above the limits listed in Table 1.

Table 1. Limits for Establishing Contamination Zones

Type of Contamination	Limit of Contamination for	
	Alpha Radiation	Beta-Gamma Radiation
Airborne, μCi/cc of air	2×10^{-12}	3×10^{-10}
Surface, direct reading	300 d/m	0.25 mr/hr
Surface, transferable, d/min	30	500

In addition to the known contamination zones, operational procedures require that contamination zones be established in the following situations:

1. The area over any cell, pit, or cubicle from which the blocks are to be removed. This zone remains a contamination zone for as long as the blocks are out and until cleared by Health Physics after the blocks are in.

2. The area immediately in front of a manipulator cell when a manipulator is to be removed.

3. Any area containing contaminated equipment (for example: areas in crane bay containing shipping casks being cleaned, and the area in front of Cell 16).

D. Requirements for Entry to Contamination Zones

1. When a zone is marked as a contaminated zone, the minimum requirements are shoe covers and "CONTAMINATION" clothing--gloves are required for any work inside the contamination zones.

2. When a contamination zone requires additional protective clothing or equipment, these requirements will be posted at the zone portal, listed in the appropriate procedure, and/or entered on the Radiation Work Permit. If two sets of requirements thus posted should not agree, the one giving the higher level of protection will apply. Some of the additional items normally used are listed below in order of increasing level of protection.

Clothing	Respiratory Protection
Double gloves	Respirator
Double shoe covers	Fresh-air mask
Double coveralls	Assault mask
Cloth hood	Airline hood
Plastic suit	Airline plastic suit
Airline plastic suit	

3. Before personnel enter a contamination zone, they will see that the proper monitoring devices are available at the zone portal for contamination checking upon exit from the zone.

Contamination zones may also contain radiation zones which require additional monitoring equipment.

E. Requirements for Exit from Contamination Zones

1. Exit from a contamination zone must always be through the zone portal.

2. Contaminated clothing must be removed at the zone portal, monitored, and placed in the proper receptacle. Disposition of clothing items is as follows:

a. Items reading from 0 to 20 mr/hr are placed in the marked yellow cans or in plastic bags marked for the purpose. These are items which will be sent to the laundry for cleaning and re-use.

b. Items reading greater than 20 mr/hr are bagged separately and disposed of as hot waste.

3. After removing their contamination zone clothing (not including single coveralls or yellow shoes), personnel must either check themselves for contamination or be checked by someone else. The approved method for this check is to pass a GM survey meter probe slowly over all surfaces of the body and to note changes in the background level of the instrument. Special care should be taken in checking the hands

and feet. This check will be made at the zone portal. When the background is high (as, for example, over an open cell), the zone portal will be extended into an area of low background.

4. If found free of contamination, personnel may leave the contamination zone.

5. If contamination is found, Health Physics and/or supervision must be contacted. In general, contamination should be removed or cleaned while the person is still at the zone portal; however, this is not always practical. When someone must leave a contamination zone while still contaminated, he should put on clean shoe covers, gloves, and/or coveralls before going to the location where final cleaning is done.

NOTE: This is the only situation in which shoe covers worn in an un-posted zone is approved.

F. Removal of Equipment from Contamination Zones

1. Equipment being removed from contamination zones can be monitored in the same manner as personnel, or it can be smeared for contamination monitoring.

2. Contaminated equipment being moved from one contamination zone to another should be wrapped, bagged, or otherwise protected during transfer.

3. Equipment stored inside a contamination zone will be wrapped, bagged, or otherwise protected if its smear level is greater than 1 mr/hr.

III. Radiation Zones

A. Definition

A radiation zone is an area where control measures are established to prevent or minimize external radiation exposure to personnel.

B. FPDL Permanent Radiation Zones

1. All process cells.

2. All manipulator cells.

3. All tank farm cells.

4. Pipe tunnel.

5. Service tunnels (Main service tunnel, Cell 20, 17, 10 service tunnel, service cubicles for Cells 11, 13, and 14).

6. Cell 20 filter cubicle.

7. Cell ventilation filter pit and house.

8. Cubicle D (behind barricade) - 1st level north.

9. All sample stations (Cell 18, 19, 20, tank farm sampler).

10. W19, W20 tank area.

C. FPDL Temporary Radiation Zones

Any area may be designated as a temporary radiation zone when the radiation readings are above the limits listed in Table 2.

Table 2. Procedure for Establishing Radiation Zones

Dose-Rate Range (rems/hr)	Immediate Action	Followup Action
0.003 to 0.006	Post low-level tags if the accumulated dose to personnel may be 20 mr	Periodic review
0.006 to 1	Post warning signs or tags	Rope off the area if the accumulated weekly dose may be 1 R
1 to 3	Post warning signs or tags; rope off	Erect a barricade which will provide absolute exclusion of personnel if the accumulated weekly dose in the area may be 12 R; lock or block entrance
Over 3	Post warning signs and tags, and erect a temporary barricade; lock and/or block all entrances	

In addition, operational procedures require that radiation zones be established in the following situations:

1. The area over any cell, pit or cubicle from which the blocks are to be removed. These areas remain radiation zones until the blocks are in.

2. The area in front of a manipulator cell from which a manipulator arm is to be removed.

D. Requirements for Radiation Zone Entry

NOTE: No radiation zone should be entered unless it is absolutely necessary.

1. The signs at the zone portal must be checked and the necessary monitoring equipment must be available.

2. In zones where the use of a direct-reading pocket dosimeter is required, personnel should check the dosimeter periodically and leave the zone when the maximum approved dose is indicated.

NOTE: This regulation applies in any case, whether or not timing is being used to determine dose.

3. When timing is being used to determine dose, personnel must leave the zone when the timer alarm sounds or when the observer signals that the time limit has expired.

4. Personnel should not remain within a radiation zone any longer than necessary.

IV. Special Control Areas

A. Definition

Special control areas are established to provide additional controls during operations when the conditions might change quickly. These areas include the following:

1. The cell top area in the crane bay when any cell block is open.

2. The first floor manipulator operating area when a manipulator is to be pulled.

3. Areas 31 and 32 when a manipulator is to be pulled from Cell 18 or 19.

4. The crane bay when a Cell 20 filter is to be changed.

5. Cell 15 change room when Cell 15 door is to be opened.

6. Cell ventilation filter pit, filter house, and bypass area when a cell ventilation filter is to be changed.

B. Clothing Requirements

These areas will be posted before operations are begun. They are not contamination zones; entry and exit of authorized personnel to these areas are unrestricted as long as the proper clothing is worn. In most cases respiratory protection, shoe covers, and "C" clothing are required. Special clothing is required for passage between a control area and the contamination zone contained within the control area. When entering a contamination zone from a control area, an additional pair of shoe covers must be put on; this second pair of shoe covers will be removed at the contamination zone portal upon exit.

C. Contamination Check

When operations within a contamination zone are completed, the control area restrictions remain in effect until a contamination check has been made in the contamination zone. If there is no evidence of release of activity inside the contamination zone, the control area may be cleared. When there is evidence of activity release inside the contamination zone a spot check is made of the control area. It is then either cleared, if no contamination is found, or zoned as a contamination zone for cleaning.

V. Miscellaneous FPDL Zone Procedures

A. Removal of Sample from Sample Station

When a sample is to be removed from one of the sample stations the immediate area is posted as requiring respiratory protection. The operator taking the sample must wear rubber gloves and respiratory protection. After the sample has been pulled and the carrier has been removed from the sample station, the operator will check the sample station for contamination. If the sample station is contaminated above 100 disintegrations/minute, it will be cleaned immediately. If the contamination level on the sample station is above 1 mr/hr, the respiratory protection requirement remains in effect.

B. Doors

1. The double doors on both ends of the cell top area in the crane bay normally remain closed. They may be opened on supervisor's instructions to permit transfers of equipment.

2. The doors between areas in the FPDL are equipped with placards reading "Entrance Requirements" to which signs may be attached; these doors are also equipped with chains. This system is used to establish control areas, and each door that has signs showing special requirements will also have the chain in place across the door. Such a posting does not make the area a contamination or radiation zone. If the area is to be made such a zone, the proper Health Physics tags or signs will be attached.

3. Addition of solid chemicals through the solids addition funnels at Cells 7, 8, 13, and 14 requires respiratory protection.

FISSION PRODUCTS DEVELOPMENT LABORATORY

CELL DANGER ZONE ENTRY PROCEDURE

A. Cell Danger Zone

A Cell Danger Zone by definition is an area where high-level radiation and contamination can be expected to exist and includes the following:

1. Process Cells 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 20, 21, 22, 23, 24, 25, 26.

2. Manipulator Cells 11, 12, 13, 14, 15, 16, 18, and 19.

3. Service tunnels.

4. Pipe tunnels.

5. Tops of Cells 1 and 15 when unloading shipping casks.

B. Cell Entry Procedure

Before personnel will be admitted to a Danger Zone, the following requirements must be met:

1. A survey has been made by the Health Physics surveyor with the proper equipment for determining personnel exposure.

2. A Radiation Work Permit has been executed and signed by FPDL supervision and a Health Physics surveyor.

3. Personnel have attached to their clothing the following personnel monitor equipment:

   a. Pencil meters.

   b. Dosimeters that will be read before, during, and after entry to a cell.

   c. Film badges.

   d. Personnel radiation monitors.

   e. Film rings to be worn on the hand normally used for hand operations.

NOTE: Plastic suits with an air supply will be used in all Danger Zones except the Service Tunnels and areas removed from the Danger Zone classification by the FPDL supervisor and Health Physicists.

If a non-plastic-suit area requires a gas mask, all clothing will be taped and no part of the body shall be exposed.

4. A timing device equipped with a mechanical or electrical alarm has been set for the determined working time.

5. An observer is standing by in a location where he can render immediate aid or call for assistance if required.

6. A monitor device has been placed in the Danger Zone with an observer or an alarm unit located outside the area to monitor sudden changes in radiation level.

7. FPDL personnel who are to enter a Danger Zone have a cutie pie in their possession.

8. FPDL personnel who are to enter top access cells have a life line attached to their waist.

C. In-Cell Procedure

1. Enter cell by the shortest but safest route.

2. Do not remove anything by hand. Use 24-inch tongs, pliers, extended brushes, or any other device that will keep the hands and body in radiation fields less than 5 r/hr.

3. Place cutie pie in a position where it can be read at a glance.

4. Check all suspicious material with the cutie pie.

5. Check dosimeter periodically and leave immediately if a reading of greater than 100 mr/hr is recorded.

6. If you move a piece of material with one hand, check radiation with a cutie pie in the other hand.

7. Place all debris in hot cans or waste cans which will be removed by remote-handling methods. Do not stand or crouch near the hot can. Check can periodically with a cutie pie or place cutie pie between the hot can and your body to show changes in radiation field.

8. Work at a normal speed. Do not become excited, as this requires a greater air supply and generally means a departure from safe work habits.

D. Exit Procedure

Personnel leaving a Danger Zone shall follow the procedure outlined below:

1. When the observer or the alarm signals the end of the working time or changing radiation levels, the employee will immediately stop work and leave the cell by the shortest and safest route.

2. When the employee reaches a designated area, the observer will wash the contamination from the plastic suit with water spray.

3. The employee will then enter a Contamination Zone and remove the contaminated protective clothing. He will move from the contaminated area to a regulated area as he removes the final piece of protective clothing.

4. The employee will then be surveyed by designated personnel and his dosimeter reading recorded on the Radiation Work Permit.

5. The employee will proceed with any personal decontamination required and will not be assigned further duties until he has met all the requirements concerning body contamination.

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