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SAFETY ANALYSIS OF BUILDING 3028

R. W. Schaich

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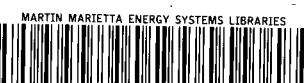
SAFETY ANALYSIS OF BUILDING 3028

R. W. Schaich

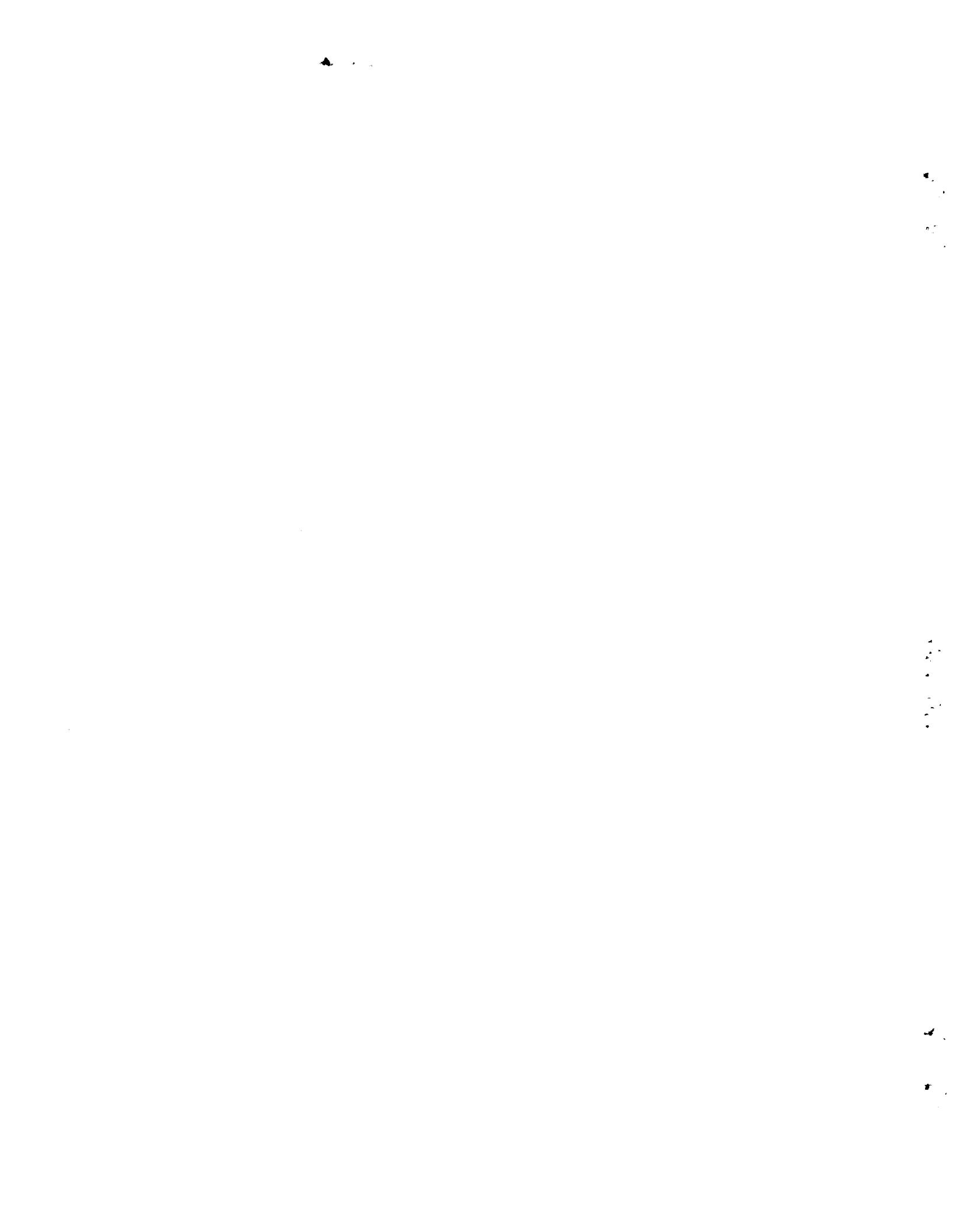
Isotopes Development Center

DECEMBER 1970

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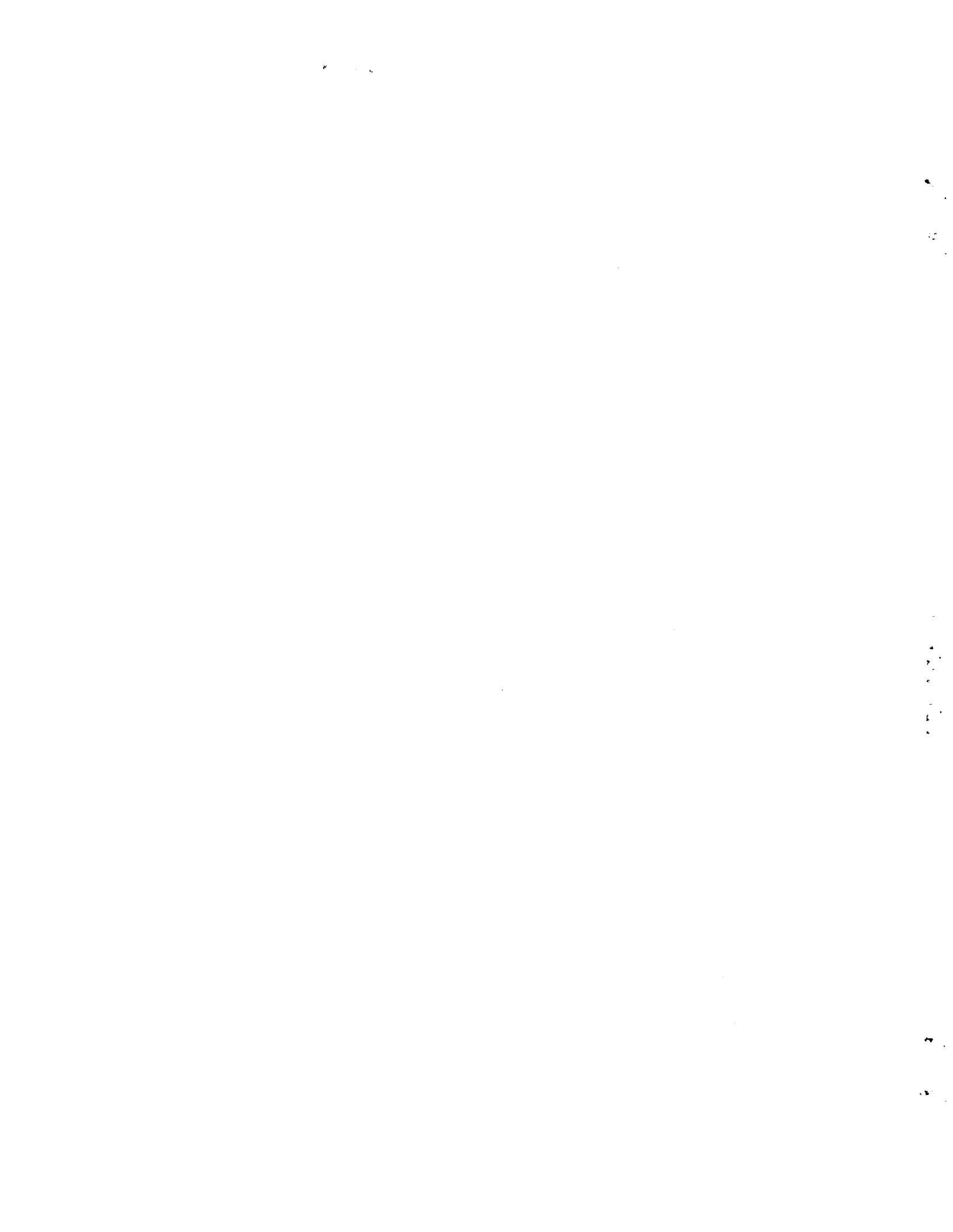


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CONTENTS

	Page
ABSTRACT	1
INTRODUCTION	1
BUILDING DESCRIPTION	2
FACILITIES AND EQUIPMENT	6
Curium Source Fabrication Facility	6
Short-Lived Fission Products Facility	9
Radioisotope Target Laboratory	11
CONTAINMENT	16
OPERATIONS	16
Curium Source Fabrication	16
Short-Lived Fission Products	19
Radioisotope Target Fabrication	23
WASTE DISPOSAL	24
Liquid Waste	24
Gaseous Waste	25
Solid Waste	25
OPERATIONAL HAZARDS	25
PERSONNEL PROTECTION	29
TRAINING PROGRAM	30



SAFETY ANALYSIS OF BUILDING 3028

R. W. Schaich

ABSTRACT

A critical review of the hazards associated with the operations performed in Building 3028 is presented. The physical facilities, operations, and operating procedures for the building are described. Analyses were made of the operational hazards and it is shown that the preliminary and secondary features of the facility are adequate to contain the radioactive materials within the confines of the building.

INTRODUCTION

Three major programs of the Isotopes Development Center are located in Building 3028. These are 1) Short-Lived Fission Products, 2) Curium Source Fabrication, and 3) Radioisotope Target Laboratories. The radioisotopes involved in these operations range from the short-lived fission products (^{99}Mo , ^{131}I , ^{133}Xe , etc.) to the transuranium elements ^{244}Cm and ^{252}Cf . The preparation of the materials processed in this facility requires a variety of operations such as dissolution, evaporation, distillation, solvent extraction, ion exchange, calcination, sintering, vacuum hot pressing, vapor deposition, welding, and leak testing. Because of the multiplicity of activities and operations, Building 3028 is designed to control the release of all radioactive solids, liquids, and gases to the environment.

The maximum in-process inventory of any one radioisotope campaigned in Building 3028 is listed in Table 1.

Table 1. Maximum In-Process Inventory

	Short-Lived Fission Products (Ci)	Curium Source Fabrication (g)	Radioisotope Target Laboratory (g)
Fission Products	5000	0	0
Americium-241	0	1000	10
Americium-243	0	1000	10
Curium-242	0	50	0.1
Curium-244	0	1000	10
Neptunium-237	0	1000	10
Plutonium-238	0	1000	10
Plutonium-239	0	0	1000
Plutonium-240	0	0	10
Plutonium-241	0	0	10
Plutonium-242	0	0	10
Uranium-232	0	1	0.1
Uranium-233	0	0	1000
Uranium-235	5 g	0	1000
Uranium-236	0	0	1

BUILDING DESCRIPTION

Building 3028 is located on the northwest corner of Isotope Circle in the Isotopes Area (Fig. 1) and is adjacent to the 3039 stack area.

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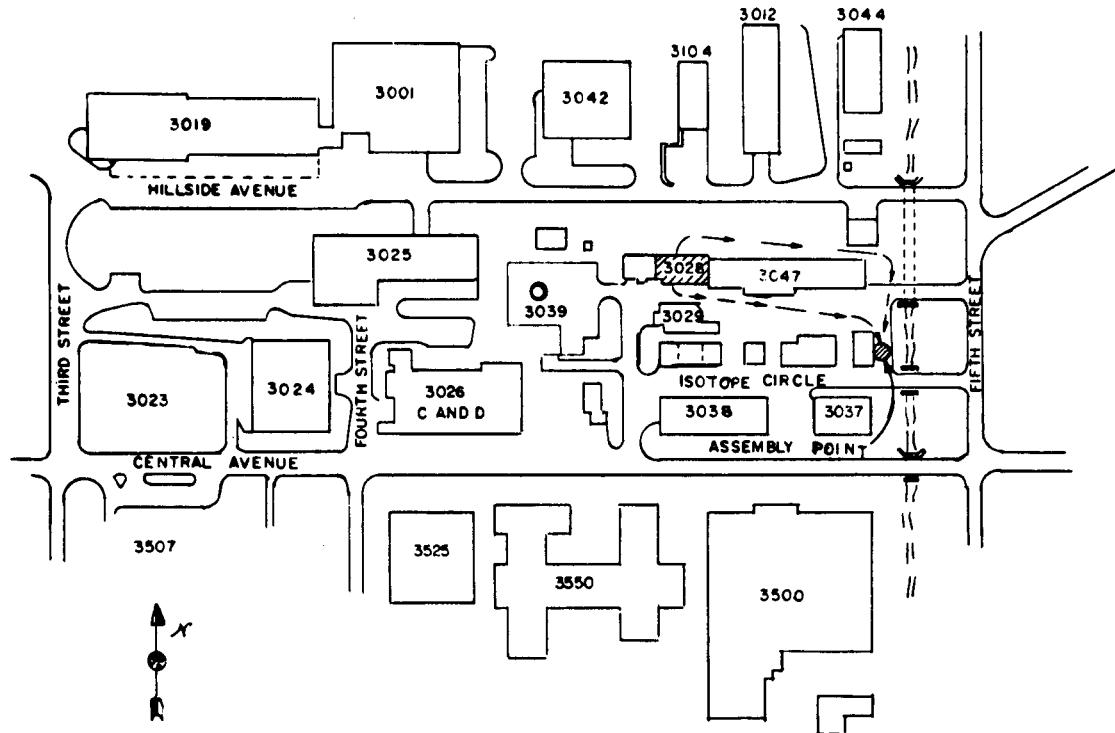


Fig. 1. Building 3028 Evacuation Route

An air lock separates Building 3028 from the Radioisotope Development Laboratory (Building 3047) on the east. There are no direct personnel accesses from Building 3028 to Building 3047. Its relation to nearby facilities in terms of distance and quantities of radioactivity normally handled is listed in Table 2.

Table 2. Adjoining Facilities and Activity Inventories

Building No.	Title	Distance			Maximum Activity Inventory (Ci)	Activity Type
		Feet	Direction			
3047	Radioisotope Development Laboratory	10	East	1×10^6	Fission Products Transuranium Elements	
3029	Source Development Laboratory	30	South	5×10^5	Fission Products	^{60}Co
3039	Gaseous Waste Area Disposal Area	100	West	0	Fission Products	
3042	Oak Ridge Research Reactor	150	North	1×10^5	Fission Products	
3030 3031 3032 3033	Radioisotope Laboratories	100	South	1×10^3	Miscellaneous Radioisotopes	
3038	Radioisotope Packing and Analytical Lab.	150	South	1×10^3	Miscellaneous Radioisotopes	
3037	Isotopes Division Offices	225	South	None	None	

The 3028 building, a steel-frame structure covered by corrugated aluminum siding, consists of a four-story operating area and a one-story cell area on the east and west sides (Figs. 2 and 3). The first floor area of the building covers 4000 ft^2 and the total volume is $80,000 \text{ ft}^3$ of free space. All areas of Building 3028 are equipped with water sprinklers connected to the ORNL fire protection system.

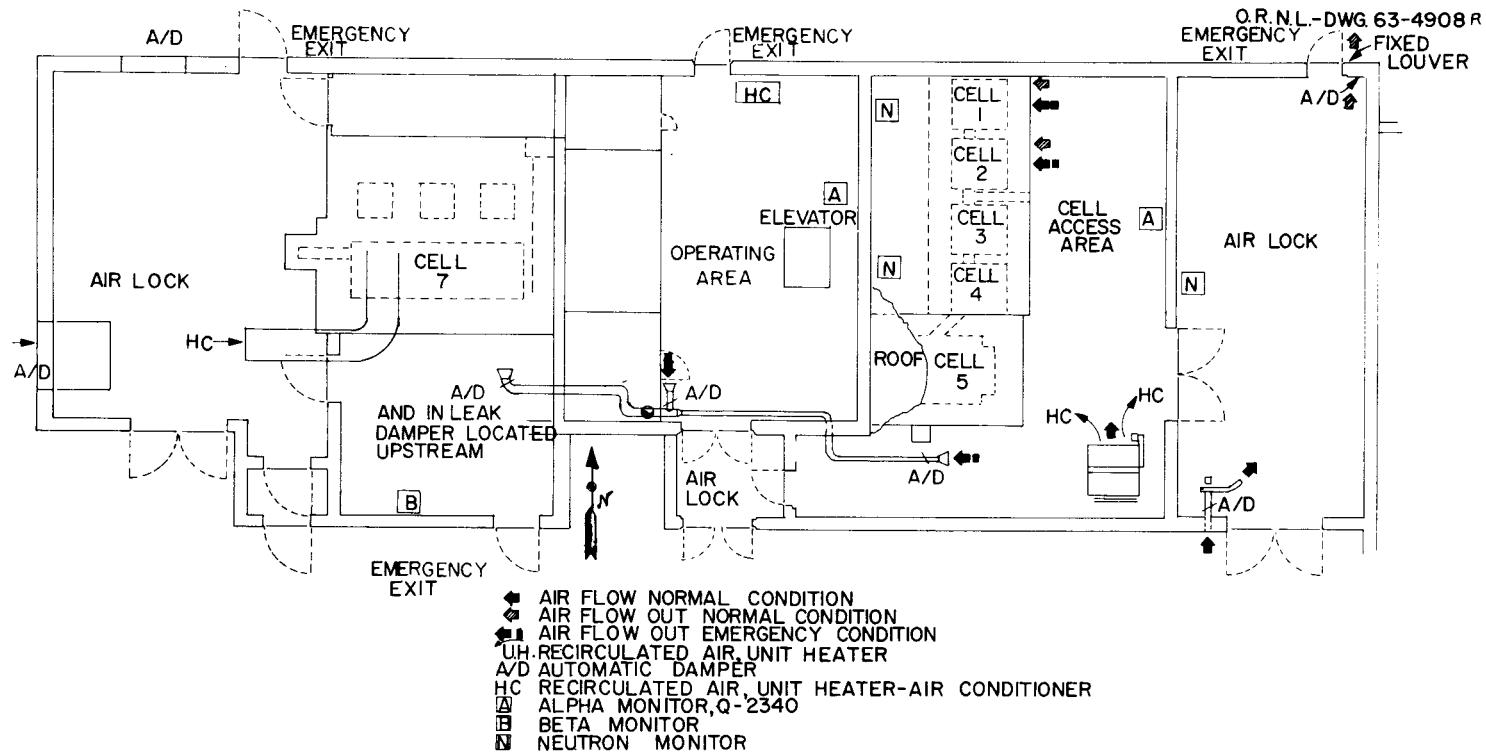
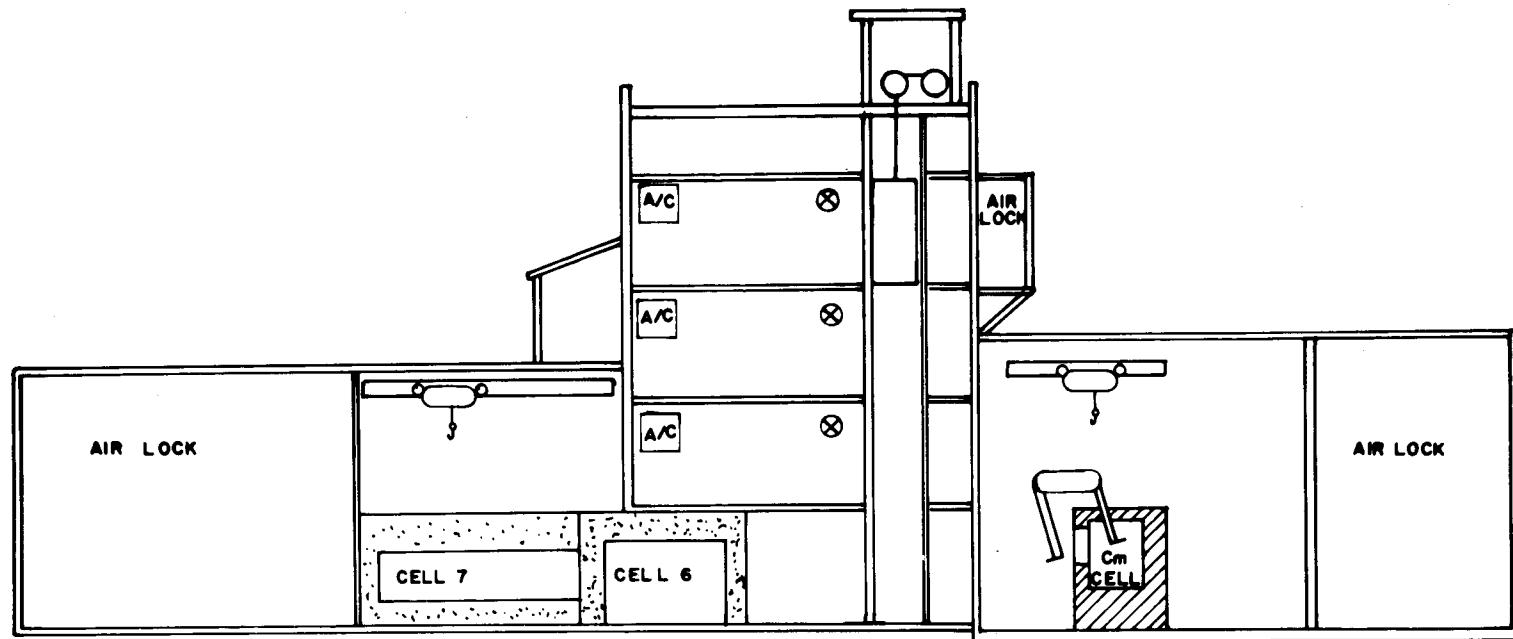


Fig. 2. Source Fabrication Facility

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⑧ ALPHA CONSTANT AIR MONITORS
A/C AIR CONDITIONER

Fig. 3. Building 3028 Cutaway

FACILITIES AND EQUIPMENT

Curium Source Fabrication Facility

The Source Fabrication Facility comprises six cells located on the first level of Building 3028 as shown in Fig. 2. The shielding characteristics and function of each cell are listed in Table 3.

Table 3. Source Fabrication Facility Cell Characteristics

Cell No.	Liner	Shielding	Designed External Radiation (mrem/hr)	Process
1	Stainless	2 ft of H ₂ O	<1	Precipitation
2	Stainless	2 ft of H ₂ O	<1	Powder preparation
3	Stainless	2 ft of H ₂ O	<1	Pelletization
4	Stainless	2 ft of H ₂ O	<1	Welding
5	Stainless	9 in. of steel + 1 ft of concrete	<1	Welding
6	Epoxy Resin	1.5 ft of barytes	<1	Off-gas cleanup

The walls of Cells 1, 2, 3, and 4 consist of steel tanks that contain 2 ft of water for neutron shielding. The steel tanks are bolted together to form the basic cell arrangement and a stainless steel liner is installed in each cell to seal all accesses to the cells. All penetrations to the cell are welded at the liner and the cells are connected in series with an air-lock system.

Extended-reach Model 8 manipulators are inserted through holes in the top of the cells which are covered by water-filled tanks for neutron shielding. The slave sections of the manipulators are covered with urethane manipulator boots sealed to the inside top surfaces of the cells. An additional plastic boot and wiper seals around the tapes in the manipulator barrels provide a secondary containment for the manipulator penetration.

Personnel access to the cells can be made by draining the water from the back shielding block and removing the tank. The cell opening is covered by a viewing window with glove ports which is gasketed to the cell

liner and can be removed for direct access to a cell. Personnel access to all Building 3028 cells is controlled by Health Physics Radiation Work Permits. All doors are sealed and locked under normal operations and are not entered until decontamination procedures have lowered radiation and contamination levels to acceptable standards.

Process Equipment

The process equipment for the Source Fabrication Facility consists of small stainless steel vessels for aqueous solution handling, stainless steel filters, furnaces for calcination of powders and pellet sintering, a vacuum hot press for pelletization, and calorimeters for thermal measurements.

The aqueous vessels are operated from a graphic panelboard, and all solution transfers are made by vacuum and gravity flow. After a solid product form is obtained, all handling operations are accomplished with extended-reach Model 8 manipulators. Due to the heat output of the ^{242}Cm and ^{244}Cm products, powder and pellet transfers are made in containers cooled with air or chilled water. The cell pans are cooled with chilled water to control cell temperatures and to remove heat in case of accidental spillage of radioactive material.

The characteristics of the vessels used in the Source Fabrication Facility are listed in Table 4.

Table 4. Source Fabrication Facility Vessels and Characteristics

Tank No.	Material Type	Volume (liters)	Agitation	Process
T-11	304 L	13	None	Feed transfer
P-11	304 L	13	Agitator	Precipitation
R-11	304 L	20	None	Vacuum receiver
S-11	Glass	0.5	None	Sampler
W-1	347	2000	Air	Waste collection
V-16	304 L	50	None	Vacuum surge tank
F-11	347	—	None	Filter
M-1	347	10	Agitator	Makeup
M-2	347	20	Agitator	Makeup

Encapsulation Equipment

Inert gas welders can be remotely operated in Cells 4 and 5 of the Source Fabrication Facility. These welders are versatile in their design

to allow the encapsulation of a variety of capsule materials. The welding is accomplished under argon atmosphere, and the capsule holder forms a chill block with water coolant to remove excess heat.

Ultrasonic cleaners can be located in Cells 4 and 5 for cleaning capsules to smear tolerances. The normal decontaminant required in the cleaning process is detergent or weak acid.

Helium leak detection equipment is operated remotely to test the encapsulated source for minute leaks.

Auxiliary Equipment

A chilled water system with a capacity of five tons is used for all in-cell cooling. The chiller consists of a 5-ton compressor unit with an automatic process water condenser. The chilled water is recirculated through the cell equipment by a dual pump system. In case of power or equipment failure, process water can be passed through the cell equipment to the process waste system on a one-pass basis. An automatic makeup tank supplies the necessary water for the chilled water system. The chilled water is sampled on a weekly basis for alpha contamination to check the process coils and jackets for leaks.

The process vacuum system services the Source Fabrication Facility cells and provides the mode of solution transfer. This equipment is located in Cell 6. A filter system of stainless steel Neva Clog prefilters and testable HEPA filters removes particulate matter before air is discharged into the Isotopes Area off-gas system. The vacuum pump is started manually when solution transfers are required. A 50-liter surge tank is located on the main vacuum header and provides an adequate catch vessel for the maximum solution volume handled in the Source Fabrication Facility cells. All vacuum transfer vessels have temperature sensitive probes which signal high liquid level in the vessel and automatically turn off the process vacuum to that vessel.

A chemical makeup area is located on the second level of the 3028 building and contains two small stainless steel vessels. These vessels are used for chemical makeup for solution addition to the in-cell vessels. The makeup tanks drain through flexible lines to funnels connected to pipes leading to vessels in the cells. The lines from the funnels have valves

located in the operating area which are opened after the funnel is partially filled with liquid. The transfer line from the funnels terminates in the cell with a stainless steel ball joint. From this point, a Tygon line is placed in the appropriate funnel for addition to the process vessel. A valve on the in-cell funnel is operated by manipulators to allow solution to flow into the vessel.

A standard wet cell battery is located in the first floor operating area to supply an emergency source of DC voltage to signal alarms in case of power failure. Emergency lighting in Building 3028 is supplied by individual wet cell lanterns, which are actuated by a failure of the 110-V AC circuit.

Short-Lived Fission Products Facility

The Short-Lived Fission Products Facility consists of one large manipulator cell with two viewing windows, a ^{133}Xe recovery and purification cell, and a low-level scrubber cell for off-gas cleanup. The main cell bank has 3 ft of high-density concrete, the ^{133}Xe cell has 2 in. of lead, and the scrubber cell has 8 in. of stacked barytes block for shielding the beta-gamma activities handled in this facility. The control of ^{131}I release is the primary design criterion from the standpoint of environmental radiation safety. The use of simple, easily replaced process equipment inside the manipulator cell and the use of manipulators for performing processing operations are the major criteria with respect to operational design.

The cell ventilation and hot off-gas services to the manipulator cell are equipped with testable HEPA filters plus testable charcoal filters (KI impregnated) with an efficiency for elemental ^{131}I of >99.9%. Since the highest potential for ^{131}I release is through the hot off-gas system, additional impregnated charcoal filters and a caustic scrubber are installed in series in the off-gas scrubber cell. All the testable filters are located in shielded areas outside the cell proper. Inside the manipulator cell, additional HEPA filters and impregnated charcoal canisters are placed over the cell ventilation outlets. These are positioned for replacement with manipulators, and procedures are provided for their transfer from the cell in shielded carriers for disposal by land burial.

Inside the manipulator cell, four glass canisters containing KI impregnated charcoal are installed in series in the off-gas system. These "home-made" canisters can be handled by manipulators and are tested before installation to show that they meet the ORNL specification of an efficiency of >99.9% for elemental ^{131}I .

Since all waste solutions from the short-lived fission products process contain some ^{131}I , control of these low-volume wastes (~10 liters per run) is necessary. This control is effected by discharging all wastes into a 1000-gallon stainless steel collection tank (WC-2) equipped with testable HEPA and impregnated-charcoal filters on the off-gas discharge. In this tank, the solutions containing ^{131}I are held in a high pH condition for decay of the ^{131}I . A remote potential for criticality exists in this vessel because of the enriched uranium in the irradiated target; therefore, depleted uranium solution is mixed with all wastes discharged to this tank.

The basic approach to process equipment design for the Short-Lived Fission Products Facility is one of simplification. The only permanently installed process vessels are a dissolver for the U-Al targets and a neutralization-distillation vessel for the ^{131}I process. The dissolver is a sealable stainless steel vessel connected directly to the ^{133}Xe recovery system through a de-entrainment trap. The neutralization-distillation vessel is a similar tank provided with fittings so that manipulator connection to various process lines can be made. Both vessels are equipped with heating-cooling jackets, pressure relief plugs, and controls for maintaining temperatures. There is a small stainless steel vacuum tank for waste holdup. All other chemical process equipment is laboratory-sized replaceable glassware.

The ^{133}Xe recovery equipment consists of a de-entrainment vessel on the off-gas from the dissolver plus molecular sieve traps cooled with liquid nitrogen or purged with helium for purification. The dissolver system is designed for minimum air leakage, and a helium purge is required to sweep the ^{133}Xe out of the dissolver and transfer it to the ^{133}Xe system.

Handling techniques and procedures have evolved as more experience with the Short-Lived Fission Products Facility has been gained. Once the

basic ^{131}I control procedures were established, it became apparent that much of the ^{131}I release problem was related to non- ^{131}I processes in which there were only small amounts of residual ^{131}I involved. Accordingly, much attention has been given to controlling ^{131}I in these systems. Reactions requiring acidic systems are carried out in closed vessels vented to off-gas through individual scrubber systems. A supply of NaOH solution is kept on hand, ready for immediate use in case of spills. During de-contamination operations, acid is used only after thorough washing with NaOH and water. Steps are taken to guard against the drying of solutions containing ^{131}I , even in the NaOH systems, since ^{131}I can be released from the dry solids.

The improvements in equipment and techniques made during the past five years have resulted in a significant decrease in the release of ^{131}I ; releases of <0.01% of total ^{131}I handled are the rule.

Radioisotope Target Laboratory

The three floor levels immediately above the cell operating area in Building 3028 have concrete floors covered with Coroseal vinyl covering, smooth wall surfaces, and adequate lighting. All three floors are air conditioned with limited makeup air.

Each floor of the target facility is isolated from the floors above or below except for removable hatches (7 by 7 ft) connecting the third and fourth and the third and first levels. These hatches are used only for movement of large equipment. Entrance to all floors is provided by a two-man elevator and air-lock entrances on the north side of the building. Emergency exit doors are provided on each floor in the southwest corner. These doors do not have air locks and, therefore, do not constitute entrances except in unusual circumstances. Each door is locked to prohibit access from the outside.

All target fabrications and associated operations using radioisotopes are performed in either steel, vacuum-inert-gas type glove boxes or Plexiglas enclosures, depending upon the type of material and the operation involved. Figures 4, 5, 6, and 7 show typical glove box installations and their locations in Building 3028.

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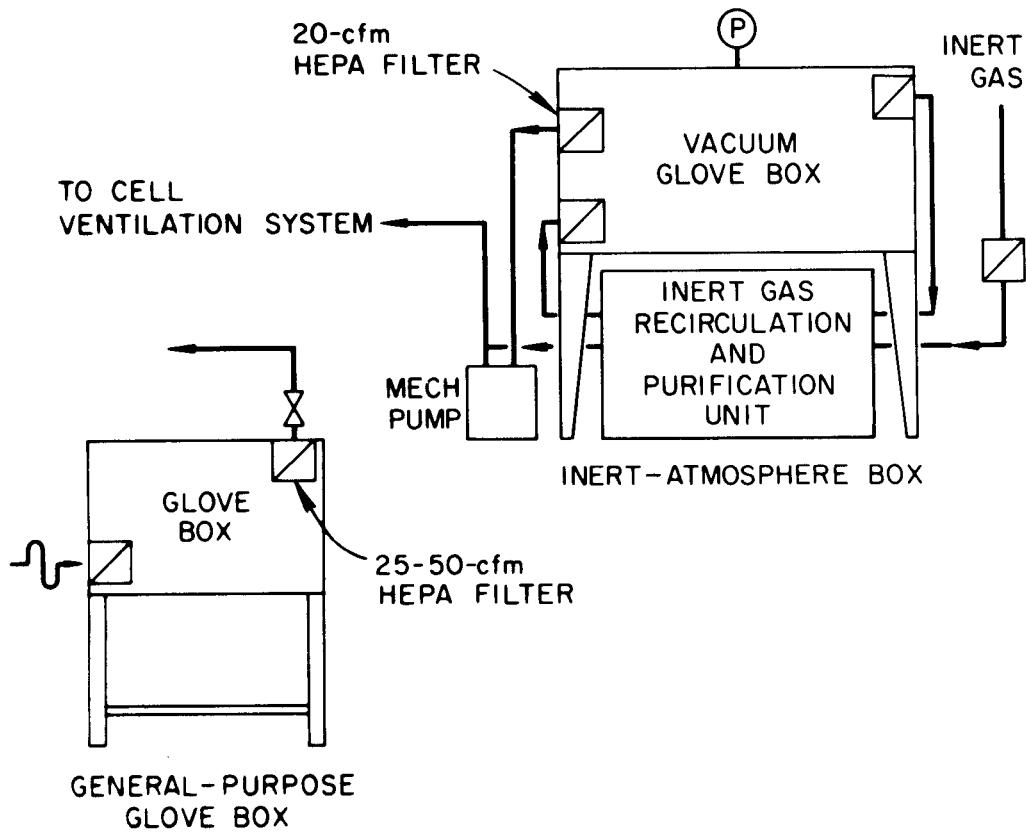
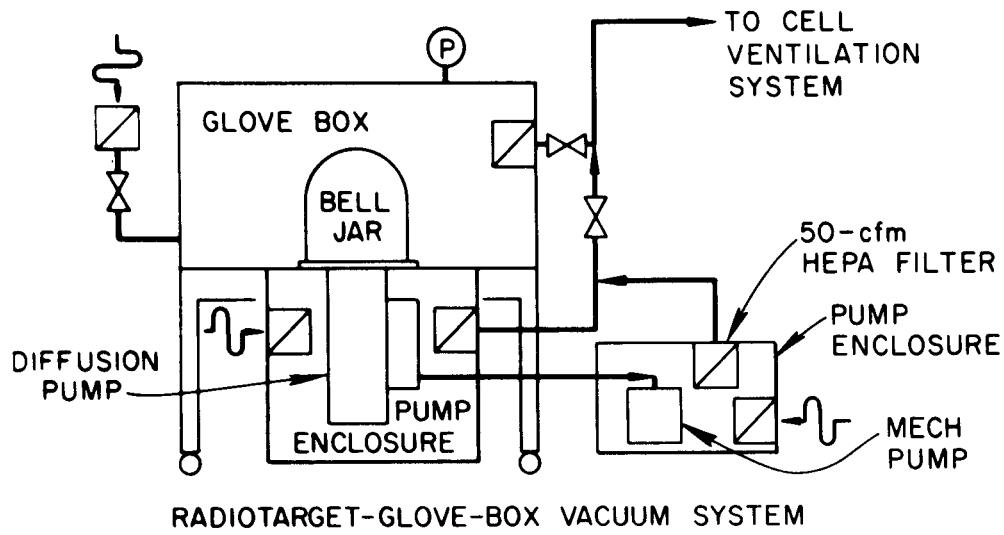


Fig. 4. Typical Glove Box Installations

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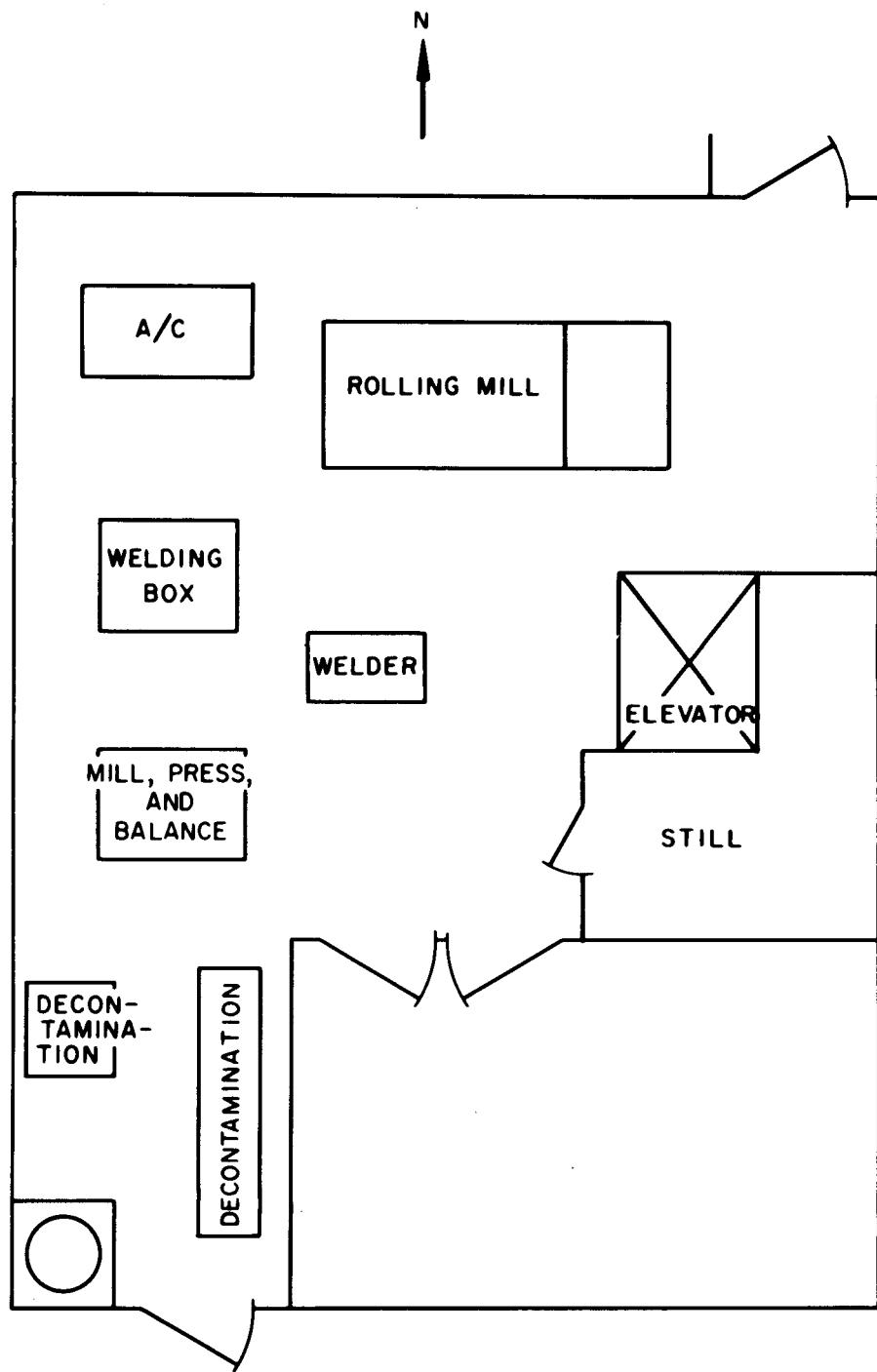


Fig. 5. Building 3028 Second Level

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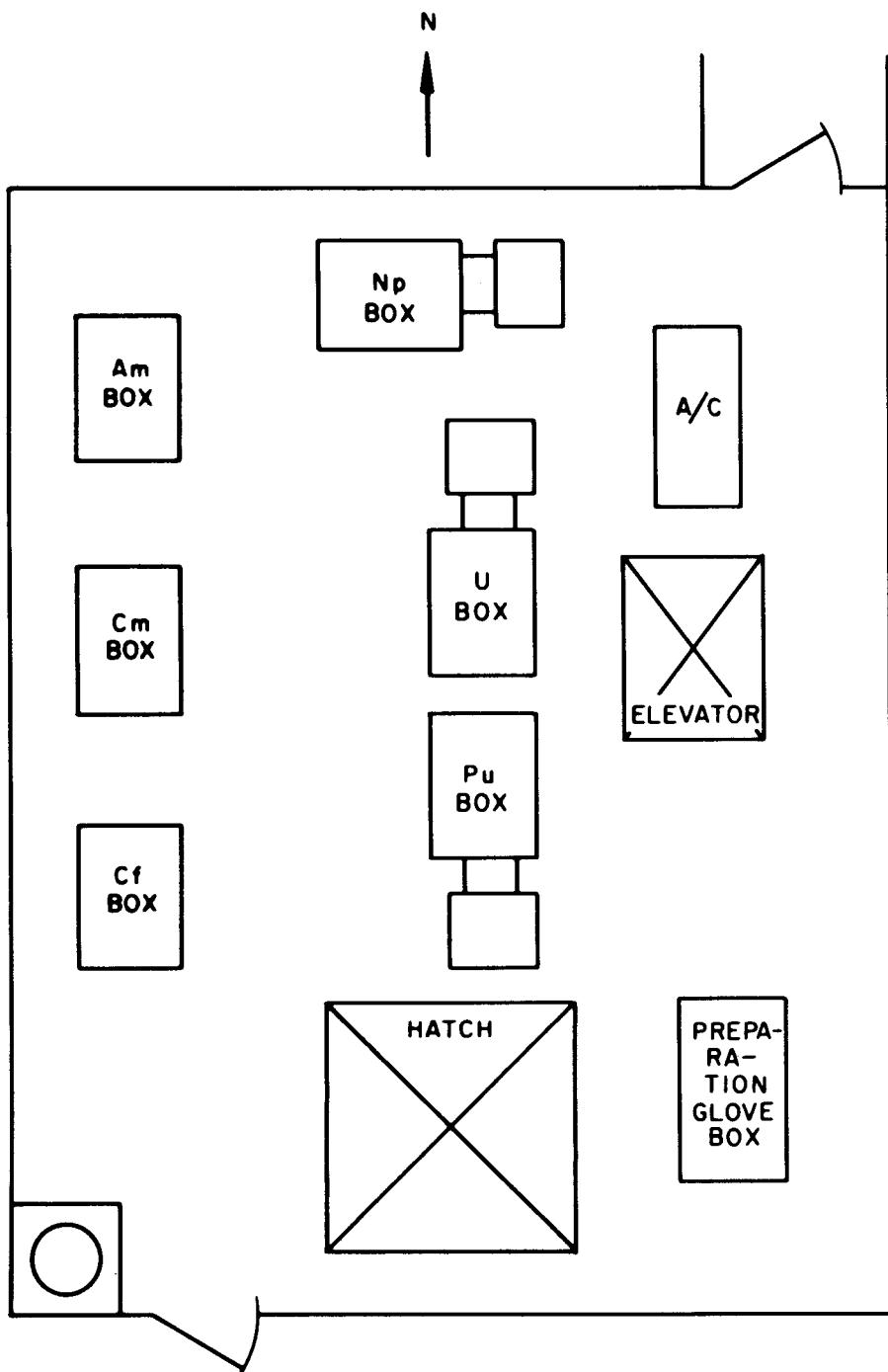


Fig. 6. Building 3028 Third Level

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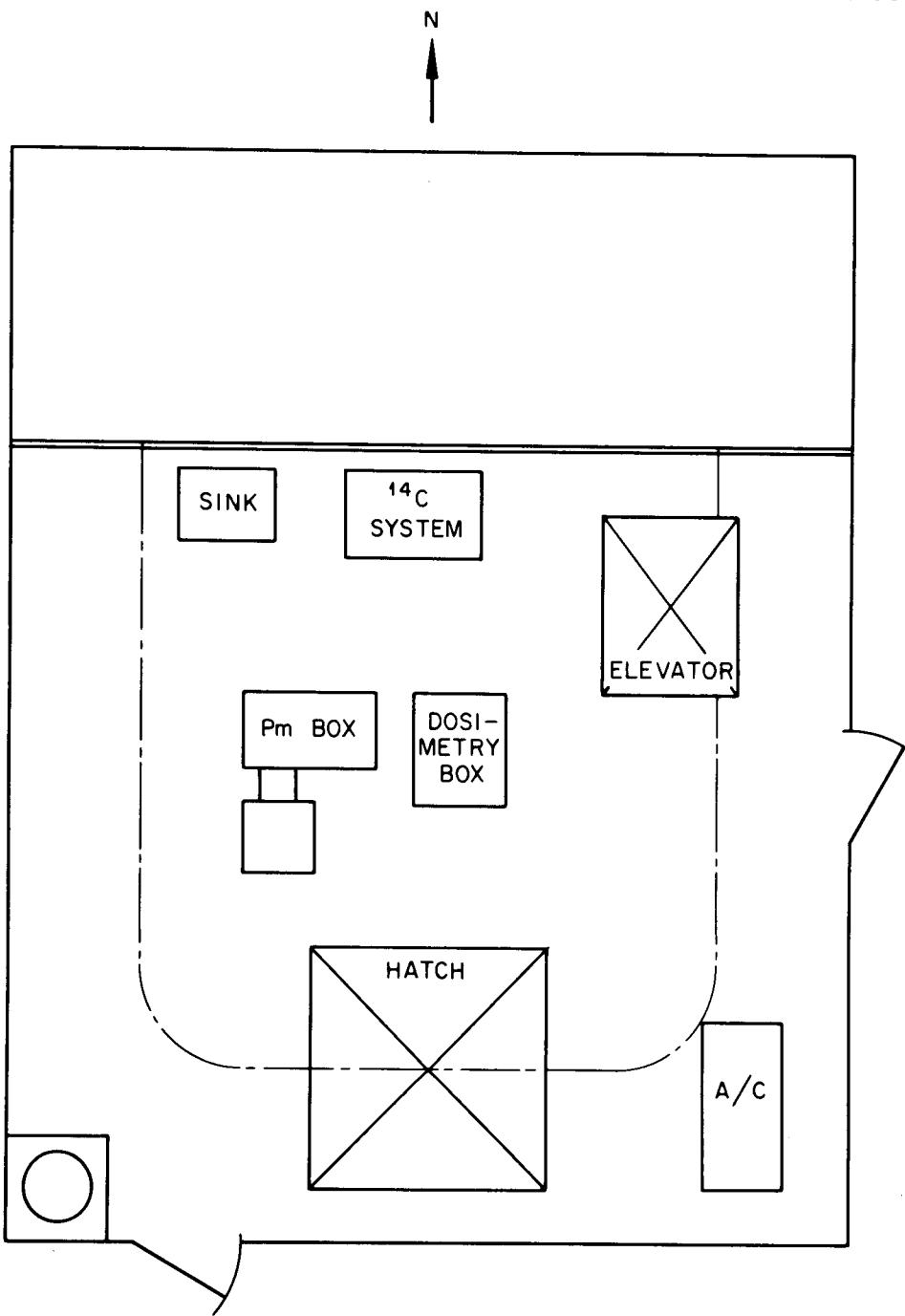


Fig. 7. Building 3028 Fourth Level

CONTAINMENT

Building 3028 (Fig. 3) was sealed by cocooning all outside surfaces of the building. All normal entries to the building have air locks for lift truck and personnel access. All doors are gasketed to reduce infiltration and are supplied with automatic closures. The building inlet air supply is limited to the building leakage rate of approximately 1000 ft³/min.

Interior partitions are provided to separate the operating areas from possibly contaminated areas, and air conditioning has been installed in all operating areas for personnel comfort.

All areas of Building 3028 are equipped with automatic dampers, discharging to the Isotopes Area cell ventilation system, to provide containment conditions of >0.30 in. of water vacuum. In the event of an accidental release of alpha, beta, or gamma activity into the building interior from the cells or glove boxes, all building openings are closed automatically. The emergency cell ventilation exhaust duct dampers are thrown to full open position and the interior of the building is exhausted to produce a vacuum of >0.3 in. of water. The signal which actuates the automatic devices is received from a one-out-of-four alarm circuitry from four alpha constant air monitors (CAM) in the alpha operating areas, a two-out-of-two circuitry in the rear of the Source Fabrication Facility cells, or a one-out-of-two alarm from two beta-gamma constant air monitors in the Short-Lived Fission Products Facility. During the containment period, the cells or glove boxes are controlled at >1.0 in. of water vacuum with reference to the building interior. All air exhausted from the building during containment is passed through roughing and testable HEPA filters before discharge to the stack. The containment signal also initiates the building evacuation horn which can be heard in all areas of Building 3028.

OPERATIONS

Curium Source Fabrication

A purified aqueous solution of ²⁴²Cm or ²⁴⁴Cm can be received at the Source Fabrication Facility in a shielded cask, which is then connected to

the Source Fabrication Facility process equipment for transfer of the solution. The unit chemical process accomplished in the Source Fabrication Facility cells is precipitation. Operations such as evaporation, decantation, filtration, and dissolution can be performed with the installed equipment.

The ^{242}Cm or ^{244}Cm can be precipitated as the oxalate in Cell 1. This cell contains two stainless steel vessels for solution transfer and one precipitator vessel. All tanks and the solution filter are jacketed and supplied with chilled water for cooling the process solution.

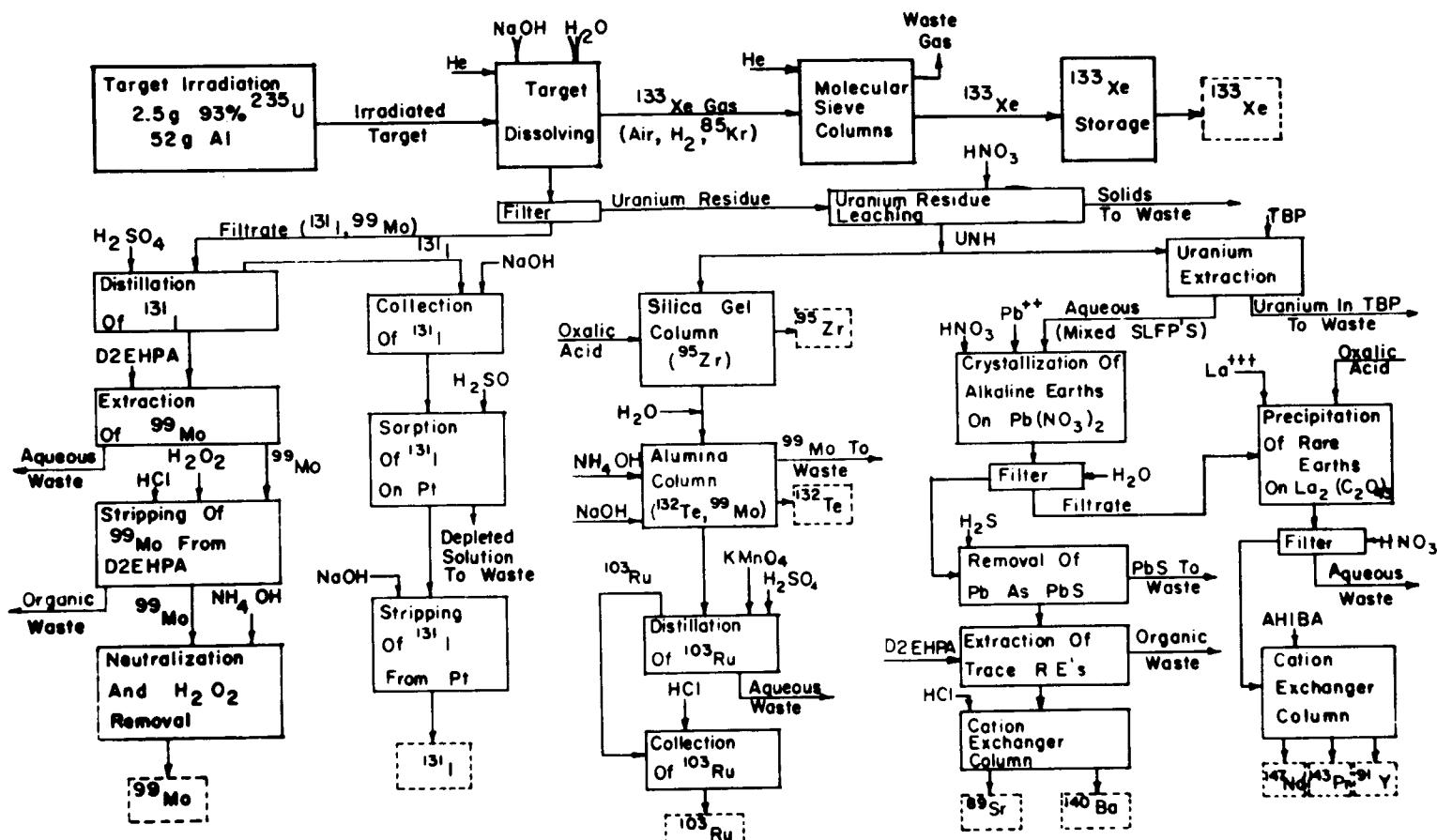
The normal operating volume of the precipitator is 10 liters and the maximum activity level in this tank is 3 g of ^{242}Cm or 30 g of ^{244}Cm . The process consists of adjusting the pH to 1.0 by adding small increments of 70% HNO_3 . A solution of 1.5 M oxalic acid is then added and the resulting solution is digested for a period of time with frequent checks on the pH. All pH determinations are made with in-cell equipment.

After digestion is complete, the slurried solution is filtered through a stainless steel filter to collect the precipitate. The filtrate is collected in a vacuum receiver tank, sampled by in-cell sampling equipment, and transferred to the Source Fabrication Facility collection tank (W-1) under the direction of supervision.

Calcination of the curium oxalate takes place in a small tube furnace at a temperature of 1000°C . After calcination, the filter is removed with manipulators and transferred to a calorimeter for product determination.

When there is no demand for ^{242}Cm , the chemical process equipment described above is used for recovery of ^{244}Cm from compounds or mixtures resulting from experiments. The Source Fabrication Facility presently receives 100-g quantities of ^{244}Cm oxide in specially designed containers from Savannah River. These containers are opened in Cell 2 and the contents are weighed and calorimetered to verify shipping data.

After calorimetric determination of the thermal output of the product, the powder is transferred to Cell 3 for blending and pelletization. The blended powder is mechanically measured into a graphite die body and placed in a vacuum hot press where the pellet is pressed under 29,000-psi pressure and 1600°C . The pelletization is continued until sufficient pellets are

NOTES:

[] Indicates Purified Material; Additional Product Conversion Required
 D2EHPA = Di 2-Ethylhexyl Phosphoric Acid; TBP = Tributyl Phosphoric Acid
 AHIBA = Alpha Hydroxy Isobutyric Acid

Fig. 8. Short-Lived Fission Products Separation

made to meet specifications concerning thermal output of the final source. These operations are conducted in argon with less than 100 ppm of oxygen in the cell atmosphere. The argon is required to prevent the formation of $^{244}\text{CmO}_2$ after the pellet material has been converted to $^{244}\text{Cm}_2\text{O}_3$.

The pellets are sealed in special capsules by remote welders located in Cells 4 and 5. These cells can be operated under argon purge with an oxygen content of <100 ppm. After the sources are helium leak tested they are decontaminated to smear tolerances and loaded into containers or generators for shipment to the customer.

Short-Lived Fission Products

The only two short-lived fission products that are routinely recovered are ^{133}Xe and ^{131}I ; the others are processed on a demand basis. The operations described are those used to provide standard products (Fig. 8).

The short-lived fission products target consists of a hollow cylinder 4.5 in. long and 1.75 in. in diameter containing 2.5 g of 93% enriched ^{235}U dispersed in 52 g of aluminum. This target is irradiated in the Oak Ridge Research Reactor at a flux of approximately $2 \times 10^{14} \text{ n/cm}^2 \cdot \text{sec}$ for an average period of 15 days. After the target has been discharged from the reactor it is transferred to the Short-Lived Fission Products Facility where it is held for two days to permit decay of very short half-life radionuclides before processing starts.

Xenon-133

The target is placed in the dissolver vessel which is connected to the ^{133}Xe recovery system. After the system has been tested for leaks, cold 6 M NaOH is introduced. The system is heated slowly until the reaction between the aluminum and the NaOH starts. This reaction is exothermic and its rate is controlled by adjusting the vessel temperature with chilled water or steam. Rare gases and hydrogen are released and are swept into the ^{133}Xe equipment with a slight flow of helium. After the reaction is complete the helium flow is maintained until all the ^{133}Xe has been removed from the dissolver.

In the ^{133}Xe equipment, the ^{133}Xe is sorbed on a molecular sieve contained in a U-tube cooled in liquid nitrogen. The ^{133}Xe is purified by

elution with helium, with the ^{133}Xe fraction first being sorbed on a second molecular sieve tube then eluted and condensed in a cold trap. The ^{133}Xe is shipped either in glass ampules or in shielded gas cylinders.

Iodine-131 and Molybdenum-99

The liquid in the dissolver is diluted to 4 M in NaOH and filtered. The filtrate contains most of the ^{131}I and ^{99}Mo plus about 15% of the ^{132}Te and traces of the other short-lived fission products. The undissolved uranium residue collected on the filter is reserved while the filtrate is processed to recover the ^{131}I and ^{99}Mo in the neutralization-distillation vessel. The filtrate solution is acidified with H_2SO_4 and then heated to distill the ^{131}I . Air is drawn through the solution under off-gas, and the air plus water vapor carry the ^{131}I to the scrubber. The ^{131}I is trapped in 1.0 M NaOH containing a trace of sulfite. When the distillation is complete, the scrubber solution is transferred to a vessel containing platinum gauze, and the solution is acidified with H_2SO_4 . The ^{131}I sorbs on the platinum, and the depleted ^{131}I solution is removed. After the platinum has been rinsed, it is contacted with 0.3 M NaOH which strips the ^{131}I from the platinum. The resulting ^{131}I solution is held as a stock from which products are prepared by adjusting the pH to between 7.0 and 9.0 with H_2SO_3 before shipment.

The liquor remaining in the neutralization-distillation vessel after the ^{131}I process contains ^{99}Mo , significant amounts of ^{131}I and ^{132}Te , and traces of other short-lived fission products. This acid solution is transferred to a batch contactor where the ^{99}Mo is extracted into a solution containing 20% di(2-ethylhexyl)phosphoric acid in an inert diluent. The phases are separated and the organic phase is washed with 1.0 M HCl; then the ^{99}Mo is stripped from the organic phase with 0.1 M HCl-2% H_2O_2 . After treatment to remove H_2O_2 , the ^{99}Mo solution is adjusted to 1 M in HCl and an identical extraction sequence is done using new equipment. The ^{99}Mo solution from the second extraction cycle is adjusted to 1 M NH_4OH as product.

Single shipments of less than 3 Ci are made as solution. For larger shipments the product solution is adjusted to pH 4-5 with HCl and passed

through a bed of alumina contained in a glass column. The ^{99}Mo sorbs on the alumina which is rinsed, dried, and shipped as a solid.

The solution from which the remaining products are recovered is obtained by leaching the uranium residue with 6 M HNO_3 . The resulting solution is divided into fractions for recovery of the various products. These may be grouped into two types - those recovered directly from the solution and those requiring preliminary removal of the uranium.

The products recovered from the uranyl nitrate solution directly are ^{103}Ru , ^{132}Te , and ^{95}Zr . Since ^{95}Nb is later recovered from the ^{95}Zr product it is also included in this group. Although it is possible to process a given fraction of the feed solution for all of these products, in practice each product is usually recovered from a separate fraction.

Zirconium-95

The uranyl nitrate solution is passed through a column of silica gel, which sorbs the ^{95}Zr . The column is washed with 6 M HNO_3 and water; then the ^{95}Zr is stripped from the silica gel with 5% oxalic acid solution. The solution is passed through a column of cation exchange resin to remove traces of contaminants. This solution is the shipping form for the ^{95}Zr and is also the feed solution from which the ^{95}Nb is recovered. This separation is done by acidifying the solution and adding KMnO_4 . A precipitate of MnO_2 forms and carries the ^{95}Nb . After separation by centrifugation, the precipitate is dissolved in 5% oxalic acid and a similar MnO_2 precipitation is done. This is repeated several times to remove traces of ^{95}Zr , with the 5% oxalic acid solution finally being passed through a bed of cation resin to remove the manganese.

Tellurium-132

The uranyl nitrate solution is diluted to approximately 2 M HNO_3 and passed through a column of alumina, which sorbs the ^{132}Te and traces of ^{99}Mo . The alumina is washed with water and NH_4OH to remove the ^{99}Mo ; then the ^{132}Te is stripped from the column with 3 M NaOH . Shipments are made either as solutions or with the ^{132}Te sorbed on small beds of alumina.

Ruthenium-103

The feed solution is adjusted to 10 M in H₂SO₄ and an excess of KMnO₄ is added to oxidize the ¹⁰³Ru. The solution is heated to distill the volatile RuO₄, which is trapped in concentrated HCl in a series of scrubbers. The scrubber solution is adjusted to 6 M HCl.

Alkaline Earths

Uranium is removed from the uranyl nitrate solution by extraction into tributyl phosphate. The ⁹⁵Zr is usually removed at this point by sorption on silica gel even if the ⁹⁵Zr is not to be recovered as product. Lead carrier is added; then fuming nitric acid is added until the lead nitrate crystallizes from the solution, carrying the alkaline earths ⁸⁹Sr and ¹⁴⁰Ba. The solution is filtered.

The lead nitrate crystals are dissolved in water, and the lead is precipitated as the sulfide and filtered. Traces of rare-earth contaminants are removed from the filtrate by extraction into di(2-ethylhexyl)-phosphoric acid. The alkaline earths are then sorbed onto a column of cation exchange resin and separated by elution with 2 M HCl. The purified ⁸⁹Sr and ¹⁴⁰Ba fractions from this elution are diluted to 1 M HCl as products.

Rare Earths

The filtrate from the crystallization of the alkaline earths with lead nitrate contains the rare earths ⁹¹Y, ¹⁴⁷Nd, and ¹⁴³Pr. This solution is taken to dryness; then the solids are dissolved in water and lanthanum carrier is added. The lanthanum is precipitated as the oxalate, carrying the other rare earths. After the precipitate has been filtered and washed, the rare earths are converted to the nitrate form and taken up in 0.2 M HNO₃. The solution is loaded onto a cation exchange resin in a column which can be heated and also can be operated under pressure. The rare earths are separated by elution with 0.3 M alpha-hydroxyisobutyric acid at pH 4.5 under a pressure of 750-1000 psi at 70°C. The purified fractions of ⁹¹Y, ¹⁴⁷Nd, and ¹⁴³Pr are acidified; then the rare earths are sorbed on individual small columns of cation resin. After these columns have been washed, the products are stripped from the resin with 6 M HCl, which is diluted to 3 M HCl as product.

Radioisotope Target Fabrication

In the preparation of metals which are easily oxidized (e.g., U, Pu, Np, and Am), inert-gas-filled glove boxes are provided. Similarly, an 8-in. warm or cold rolling mill is enclosed in a steel glove box which is constantly maintained under an argon atmosphere. All steel glove boxes have attached argon purification trains to continuously remove oxygen, nitrogen, and water vapor to levels <10 ppm. Each inlet and outlet argon recirculation line is fitted with canister-type HEPA cartridges to prevent contamination from entering the argon purification systems.

Evaporation systems generally employ oil-diffusion-pumped or ion-pumped Pyrex glass chambers. An electron-bombardment heat source similar to those described in the Target Preparation Center annual report¹ is employed to evaporate materials such as NpO₂ and UO₂. Since these systems are under high vacuum during evaporation-condensation processes, the vacuum chamber provides a third containment zone. Each target preparation requires specific procedures because rarely is more than one target made to the same specifications. Because of this, a production-type routine operations flow-sheet cannot be logically described; however, in the annual report noted, complete procedural outlines are given for several different materials. All of these glove boxes contain air atmospheres, and each box has a continuous flow of air with testable HEPA filters at the inlet and outlet. This flow is maintained by a vacuum of >0.5 in. of water created by direct connection to the cell ventilation system.

Rolling of alpha-emitting metals is preceded by conversion of the metal oxide to metal by simultaneous reduction-distillation as described in ORNL-3829. Reduction of oxides of neptunium, americium, and curium is performed using thorium metal as the reductant with subsequent distillation of the product metal. Curium dioxide is prereduced to the sesquioxide to avoid spontaneous reaction between the dioxide and thorium metal. In all cases transfer of oxide or metal samples between glove boxes is made in stainless steel containers. Radiofrequency heating is used to generate the required temperature. Similarly, alloys of these metals are prepared by fusion of the components in vacuum or an inert-gas atmosphere. Usually

¹E. H. Kobisk, Review of Isotopes Target Program October 1963 - December 1964, ORNL-3829, Oak Ridge National Laboratory (September 1965).

small quantities of these radioactive metals are employed - between 50 and 100 g. If quantities larger than 100 g of metal are required, this material will be furnished in metal form by the customer.

Metals to be rolled are usually inserted in stainless steel sandwiches so that the sample does not directly contact the rolls; both open and welded sandwiches may be used. If welding is required, provisions for the requisite equipment are available in one of the inert-gas steel boxes. A preheat furnace for use in warm or hot rolling is included within the glove box enclosure surrounding the rolling mill. The metal (encased in stainless steel) is preheated, passed through the rolls, and returned to the furnace for reheating.

WASTE DISPOSAL

Three types of waste result from operations in Building 3028: liquid, gaseous, and solid. The sources from which these wastes derive and the means of disposing of them are described below.

Liquid Waste

The liquid wastes in Building 3028 consist of intermediate level waste from cells, process waste from building floor drains, and storm sewer waste.

All cell wastes are sent to tank WC-2 or WC-10 on the ORNL Tank Farm via the cell floor drains. The normal volume of waste should be less than 50 gal/day and the normal activity level should be less than 200 mCi/gal. The WC-2 transfer system is contained for alpha waste handling and the tank is vented to off-gas through two testable HEPA filters and one testable charcoal filter. The WC-10 transfer system is contained for alpha waste handling. All waste volumes are continuously monitored by the ORNL Waste Monitoring System. The process waste from building floor drains is negligible; therefore, the only source of contamination to this system would be solutions used to decontaminate the operating area floor. This process waste is continuously monitored for alpha and beta-gamma activity and volume by the ORNL Waste Monitoring System. The storm sewer waste consists of all roof drainage and condensate from the building heating system; these are nonradioactive. The only possible contamination in this

system, therefore, would come from an outside source which settled on the building roof.

Gaseous Waste

Gaseous wastes normally originate during cell or glove box operations and exhaust to the ORNL cell ventilation and off-gas systems. Building 3028 is kept at a vacuum of greater than 0.05 in. of water by means of ducts from the cell ventilation system. All air passing through this system goes through a testable HEPA filter in Building 3028 and then through FG-25 roughing filters and testable HEPA filters in the Isotopes Area filter house before discharge to the 3039 stack. The cell or glove box atmosphere is triply filtered through HEPA filters. The primary (cell ventilation) filters are changed routinely but are not tested after initial installation. The other two sets of HEPA filters, however, are routinely tested by the Inspection Engineering Group. Off-gas from in-cell vessels is filtered through two testable HEPA filters plus testable charcoal filters for ^{131}I removal. All HEPA filters must meet a DOP test of greater than 99.95% efficiency for particles greater than 0.3 micron.

Solid Waste

All solid wastes from Building 3028 are handled according to the Isotopes Division Solid Waste Handling Procedure.² All alpha solid waste is considered as high-level waste. All materials are washed and removed from the cells or glove boxes by standard bagging techniques or bottom loading shielded containers. The material is triply contained and checked before being transported to the Solid Waste Handling Facility.

OPERATIONAL HAZARDS

Three of the Curium Source Fabrication cells were designed for inert atmosphere operations with an oxygen content of less than 100 ppm. The cell liners, electrical penetrations, services, etc., are sealed with vacuum-type fittings to prevent the inleakage of air. If a pressure or vacuum of >10 in. of water is accidentally created within the cell liner, the cell window will fracture and break the primary containment barrier of

²J. H. Gillette, Isotopes Division Solid Waste Handling Procedure, Oak Ridge National Laboratory (April 1, 1968) (For internal use only).

the cell. To prevent this type of accident, a photohelic cell will control the cell atmosphere to a water vacuum of 1.0 in. If the cell exceeds a water vacuum of 2.0 in., the photohelic cell will energize an alarm circuit and increase the flow of air or argon to the cell to maintain the water vacuum of 1.0 in. These cells are connected to the Isotopes Area cell ventilation system, which has a maximum water vacuum of 4.0 in. Thus a failure of the control circuitry could not create a negative pressure great enough to fracture a viewing window.

All equipment installed in the cells requiring additional gas flow will be safeguarded by reducing stations and orifices to prevent an over-pressure situation. If a vacuum system is attached to the cell, the discharge capacity of the system will be limited to <5 ft³/min. The cell atmosphere control system is designed to automatically meet this flow condition and hold the cell at 1.0 in. H₂O vacuum. Inlet gas flow indicators are mounted on each cell and pressure drop readings in the in-cell HEPA filters will be recorded on a daily basis to determine the proper gas flow to each cell. The photohelic control system is designed to be fail safe in case of electrical power failure by turning off all flow to the cells. The ORNL stack ventilation system is fail safe by the automatic switching to steam turbines to supply power to the cell ventilation and off-gas fans.

The measurement of the high-temperature (up to 2300°C) chemical and physical properties of radioisotope power source materials is typical of a category of operations which may from time to time be carried out in the source fabrication cells. The measurements can include surface tension, viscosity, emissivity, heat capacity, compatibility, vapor pressure, melting point, thermal conductivity, and helium release. All of the above measurements require the attainment and maintenance of high temperatures.

A failure of the cooling water to a furnace which is operating at a power level of several kilowatts could result in a heat load in excess of the dissipation capabilities of the hot cell. The cooling water outlet is equipped with a flow detector which will open a switch in the event of a reduction in flow of the cooling water. The switch de-energizes a breaker which removes power from the furnace power supply and opens the normally closed cooling water valve. The pressure in the hot zone of the furnace

is monitored by a pressure-sensitive switch which in the event of a water leak into the hot zone of the furnace would perform the same functions as the flow switch.

Hydrogen will be used as a reducing agent in some experiments. The explosive limits of hydrogen at atmospheric pressure and room temperature in oxygen are 4.65 to 93.9% and in air 4.00 to 74.2%. An analyzed mixture of 4% hydrogen-96% inert gas will be used as a preventive measure, and experiments of this type will be accomplished in cell atmospheres containing <100 ppm oxygen. These cells are continuously monitored by a Beckman oxygen analyzer.

The encapsulation of radioisotopes in stainless steel or other suitable metal containers will be performed by welding samples in appropriate containers. Since this is performed in an inert gas cell or glove box, the arc itself is essentially inert except that it is a source of high temperature. During welding (especially of massive materials), chill blocks will be used to avoid large temperature increases within the cell and no solvents will be permitted within the cell during such operations. Since argon or helium purges are employed through the torch head, the possibility of increasing operating pressure exists, but this problem is overcome by employing a maximum pressure photohelic control which will provide additional cell exhaust capability through use of cell ventilation facilities.

In the Radioisotope Target Laboratory, vacuum evaporation and condensation of thin films consisting of alpha- and neutron-emitting isotopes is employed. It is conceivable that some material may be exhausted through the pumps and into the cell ventilation or hot off-gas systems. Past experience with this type of evaporation has shown such occurrences to be nearly impossible because of the scrubbing action of the oil vaporization pump and the oil-containing rotary pump. The possibility of plugging HEPA filters in the facility with oil vapors originating from the vacuum pumps is eliminated by the installation of de-misters in the exhaust headers from the pumps. These devices will also assure further protection from the possibility of exhausting radioactive material from the pumps.

Preparation of target films, foils and massive shapes requires the rolling of metal, compacting (by pressing oxide powders), or vacuum evaporation-condensation of oxide or metal. When rolling operations are performed for ^{233}U , ^{235}U , or ^{239}Pu , use is made of the 8-in., 4-high glove boxed mill on the second floor of 3028. This is an entirely dry operation in an argon atmosphere. Materials to be rolled are annealed in the enclosure, stored under vacuum or argon within the box (a maximum of 1 kg), and rolled at room temperature. An argon atmosphere is required to prevent oxidation of metals during processing. Foil thicknesses between 0.020 in. and 0.0002 in. are formed starting with massive (usually 200 to 400 g) slabs of arc-melted and drop-cast material.

In two steel vacuum-inert atmosphere glove boxes, also located on the second level of 3028, weighing of small quantities (usually less than 10 g) of metal and oxide forms of these fissile isotopes is performed in preparation for canning in approved stainless steel shipping capsules. Subsequent closure of the capsules is made by TIG welding of the containers. Decontamination of external surfaces of the capsules is performed by ultrasonic cleaning with an aqueous detergent solution outside these glove boxes. These encapsulations are limited to ^{233}U and ^{239}Pu in quantities <200 g. However, at any given time 1 kg of material might be contained within a glove box from which smaller quantities might be dispensed. No water, oil, or organic materials (other than gloves) are permitted in these enclosures because of the necessity of maintaining inert atmospheres.

Vacuum evaporation of the three isotopes as metal or oxide is performed by standard procedures using electron bombardment heating and refractory metal crucibles, such as tungsten. This is a dry operation usually involving a gram or less of charge material. Three glove boxes isolated from one another have been fitted with glass vacuum chambers in which this operation is performed. Cooling water is circulated through the electron bombardment "gun" during operation. A separate Plexiglas glove box is attached in tandem to each of the steel boxes containing the evaporation systems. Each appendix box contains a balance for determining charge weights and film deposit thickness. Each pair of attached glove boxes is designated to handle a specific element and cross-contamination is avoided.

All of these systems are located on the third floor of 3028. Storage of about 100 g maximum of ^{233}U oxide and ^{239}Pu oxide for use in this operation is handled in an isolated steel glove box on the same floor but at about 10 ft from the evaporation boxes. No Plexiglas is used in this box or in its vicinity.

Administrative control of all material movement (in approved birdcage or other shipping containers) is exercised; since all operations on the second and third floors of 3028 are the sole responsibility of the Radioisotope Target Group and no other personnel are involved, such control is highly efficient. Entrance and egress of materials into all glove box facilities noted above is performed by bag-in, bag-out procedures using plastic (vinyl) bags over entrance ring ports.

PERSONNEL PROTECTION

The personnel protection program at Building 3028 is implemented through operating safeguards, radiation and contamination detection devices, training of personnel, preventive maintenance, and a continuing interest in maintaining a high safety standard.

The chemical safety practices specified in Section 1.4 of the ORNL Safety Manual are followed and special regulations that are applicable to certain jobs are posted.

Emergency cabinets are maintained in each section of the building for protection of personnel. These emergency cabinets are stocked with coveralls, gas masks, shoe covers, gloves, flashlights, and rope. All employees know the location of these cabinets and their contents.

The cell shielding is designed to limit the beta-gamma-neutron dose to personnel to <1 mrem/hr. The exposure from normal operations does not exceed 50 mrem/week. The greatest amount of exposure will come from de-contamination work. The hand exposure in glove box operations is monitored by Health Physics personnel using film rings to control the radiation dose to personnel.

Radiation and contamination detection devices are located throughout the building. Monitrons (3 beta-gamma and 4 neutron) are located at strategic points in the building for the detection of beta-gamma-neutron

radiation, and all monitors are connected to an alarm and panelboard system located in each operating area and the main entrance to Building 3028. Monitors are also equipped with local alarms to notify personnel of hazardous conditions. All monitron units are checked daily for operability by Health Physics personnel. Building 3028 is connected to the ORNL central monitoring system.

Alpha constant-air monitors and beta constant-air monitors are strategically located in all operating and cell areas. These instruments are equipped with local and central alarms and are checked for operability daily.

Portable alpha counters, G-M survey meters, cutie pies, and neutron instruments are available for use in the building, and all operating personnel are trained in the use of these instruments.

Alpha and beta-gamma survey meters are located adjacent to the normal exits of the operating areas; all personnel are required to check themselves for contamination before leaving the building. Portable instruments are also available for checking alpha, beta, and gamma contamination on the outer garments.

Radiation detection equipment is located in the Health Physics Office in Building 3047 - an alpha smear counter, a beta-gamma smear counter, high-level radiation probes, portable air samplers, Hi-Vol air samplers, cutie pies, neutron and alpha survey meters, and G-M survey meters. All operating personnel are trained in the use of this equipment. Failure to use the radiation detection instruments under specified conditions is grounds for disciplinary action. All instruments are on the Instrumentation and Controls Division programmed maintenance list and are checked and calibrated on a regular basis. An emergency evacuation alarm is connected to the containment system and can be heard in all areas of the building.

TRAINING PROGRAM

Although there is no formal training (i.e., classrooms, lectures, etc.) of the personnel in Building 3028, all of them are thoroughly familiar with the standard operating procedures of the Division regarding safety, contamination control, radiation exposure, and manipulator cell

operation. In addition, all personnel are thoroughly trained in the use of radiation instruments, and a record of their training is kept.

Some of the personnel who normally work in the building are members of the Isotopes Area Emergency Squad³ and are given training in fire prevention and first aid.

³Radioisotope Area Emergency and Evacuation Procedure, Oak Ridge National Laboratory (January 1970) (For internal use only).



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