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SUBJECT: Hazards Report for Building 3517 (Fission Products Pilot Plant)

TO: J. H. Gillette

FROM: T. A. Butler, E. E. Beauchamp, Eugene Lamb, R. W. Schaich

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ABSTRACT

A critical review of the hazards associated with the operation of the Fission Products Pilot Plant (Building 3517) is presented. The physical facilities, chemical operations, and operating procedures for the plant are described. Analyses are made of the maximum creditable accidents involving chemical explosions in radioisotope processing and a criticality incident. It is shown that the primary and secondary containment features of the facility are adequate to reduce radioisotope contamination external to the building to tolerable levels in the event of the maximum creditable accidents.

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1.0 INTRODUCTION

1.1 Purpose and Uses

The Fission Products Pilot Plant (F3P), Bldg. 3517, has been used to recover purified long-lived fission products from aqueous wastes since its completion in 1958. The future use of the F3P will be the same except that the feed material will also include fission products previously separated into chemical groups at other AEC production sites. The fission products Cs^{137}, Ce^{144}, Pm^{147}, and Sr^{90} are processed in solution and are finally precipitated as the required chemical compound, which is calcined to a dried powder and pelletized. Both gamma and beta activities in the multi-kilocurie to megacurie range are processed.

1.2 Location and Distance from Other Facilities

The F3P is located near the corner of Third Street and White Oak Avenue (Fig. 1). Its relation to nearby facilities in terms of distance and amounts of radioactivity normally processed is:

Bldg. No.	Name	Distance ft	Distance Direction	Activity Inventory, curies	Type of Activity
3517	F3P	-	-	1.2×10^6	Fission products
3508	Isolation Laboratory	125	East	Gram amounts	Transuranic elements
3505	Metal Recovery Plant	20	North	$<10^3$	Zr-Nb, Ru
2528	Thorium Oxide Plant	260	West	Negligible	ThO_2
	Tank Farm	175	North	3×10^8	Fission products
2519	Steam Plant	575	West	None	None
3504	Waste Disposal Laboratory	140	East	10^{-3}	Fission products

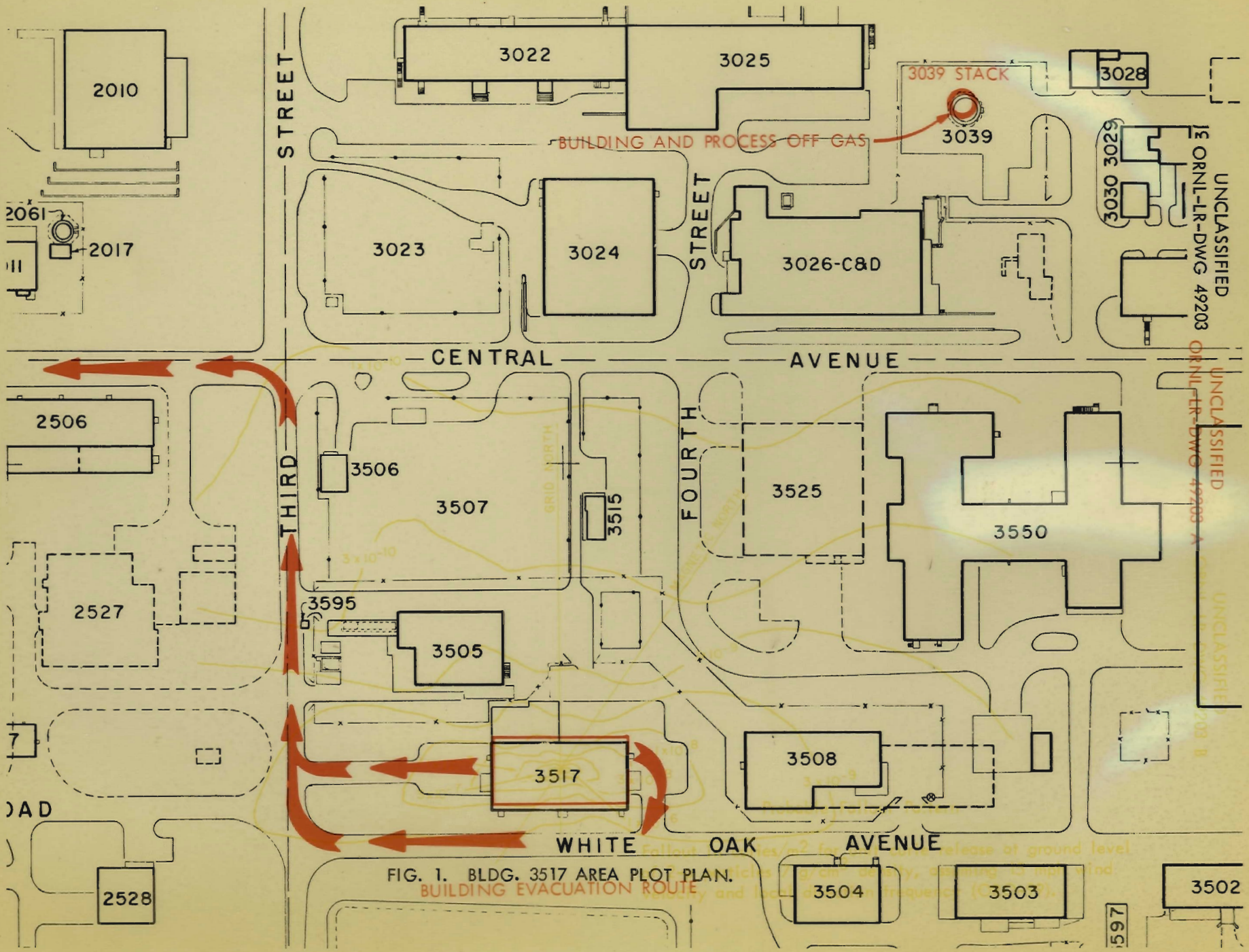

FIG. 1. BLDG. 3517 AREA PLOT PLAN.

1.3 Building Description

Building 3517 is a concrete block and corrugated aluminum-sided structure with over-all dimensions of 123 ft 6 in. long x 62 ft 8 in. wide x 61 ft 6 in. high with a total building volume of 375,700 ft^3 excluding the air locks and underground tank farm. The building roof is uninsulated steel decking with an asphalt and crushed stone topping. Interior partitions of poured concrete, concrete block, steel paneling and gypsum board are used within the building. Figures 2 and 3 show the building layout.

The main volume of primary containment within the building is the cell block, comprising 15 cells shielded with massive concrete walls (red overlay on Figs. 2 and 3). In addition, there are four other cells shielded with steel or concrete located outside the main cell block. The outer walls and roof of the building enclose the secondary containment volume (green overlay, Figs. 2 and 3). All joints are sealed with mortar or a vinyl plastic "cocoon" sprayed on a glass cloth membrane covering the joint, which forms an air seal between the building and the outside environment. All entrances to the building are by way of air locks.

1.4 Personnel Control

The numbers of people normally occupying Bldg. 3517 and adjacent facilities are:

	No. of People	
Bldg. No.	Week-Days	Nights and Weekends, per Shift
3517	12*	6
3508	12	0
2528	3	1
3505	7	3
2519	7	4
3504	25	0
Tank Farm	0	0

*Includes operating personnel and Health Physics surveyor. Additional maintenance personnel will vary widely.

The evacuation route is shown on the red overlay of Fig. 1.

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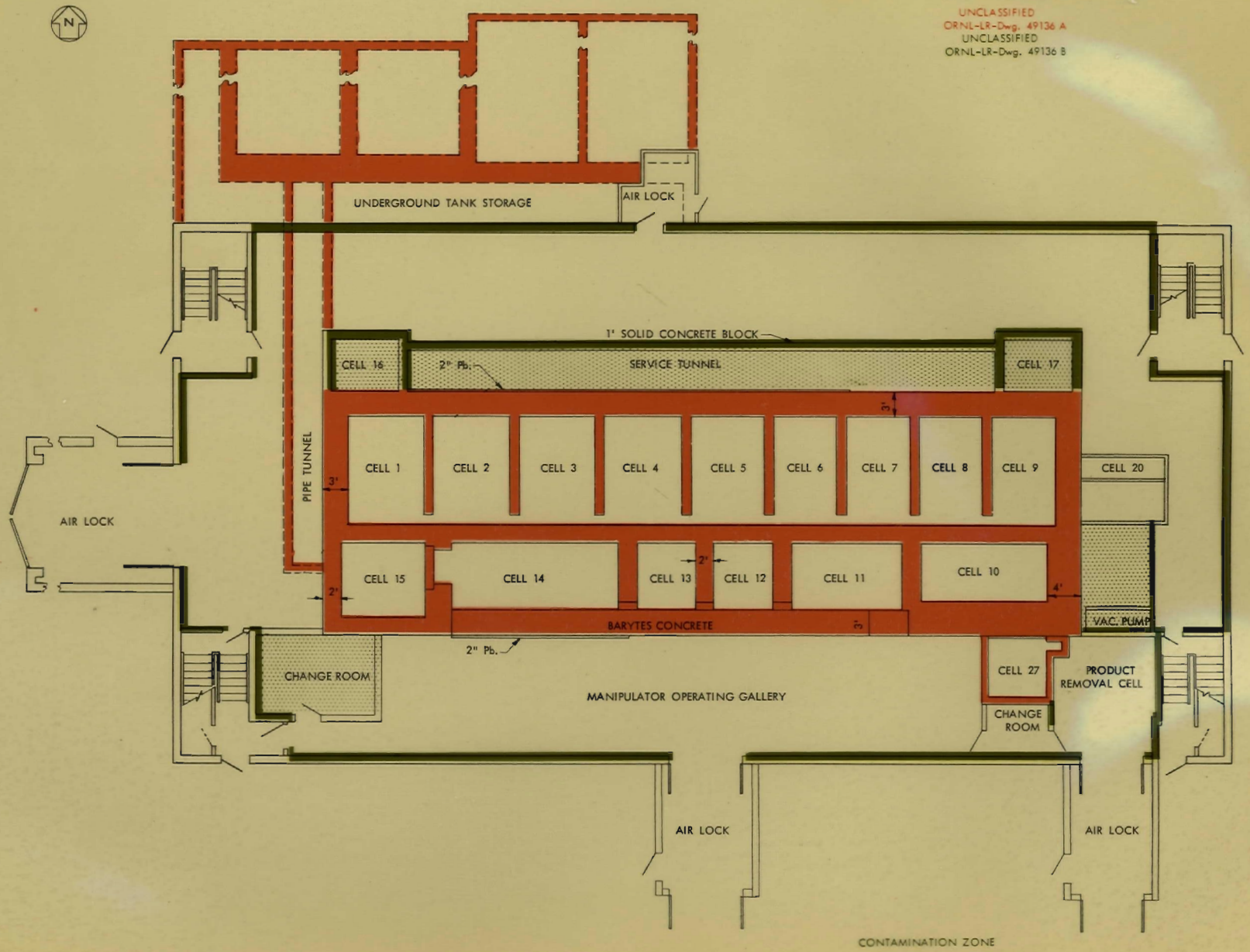

FIG. 2. BLDG. 3517 FIRST FLOOR PLAN

LIMITS OF PRIMARY CONTAINMENT

LIMITS OF SECONDARY CONTAINMENT

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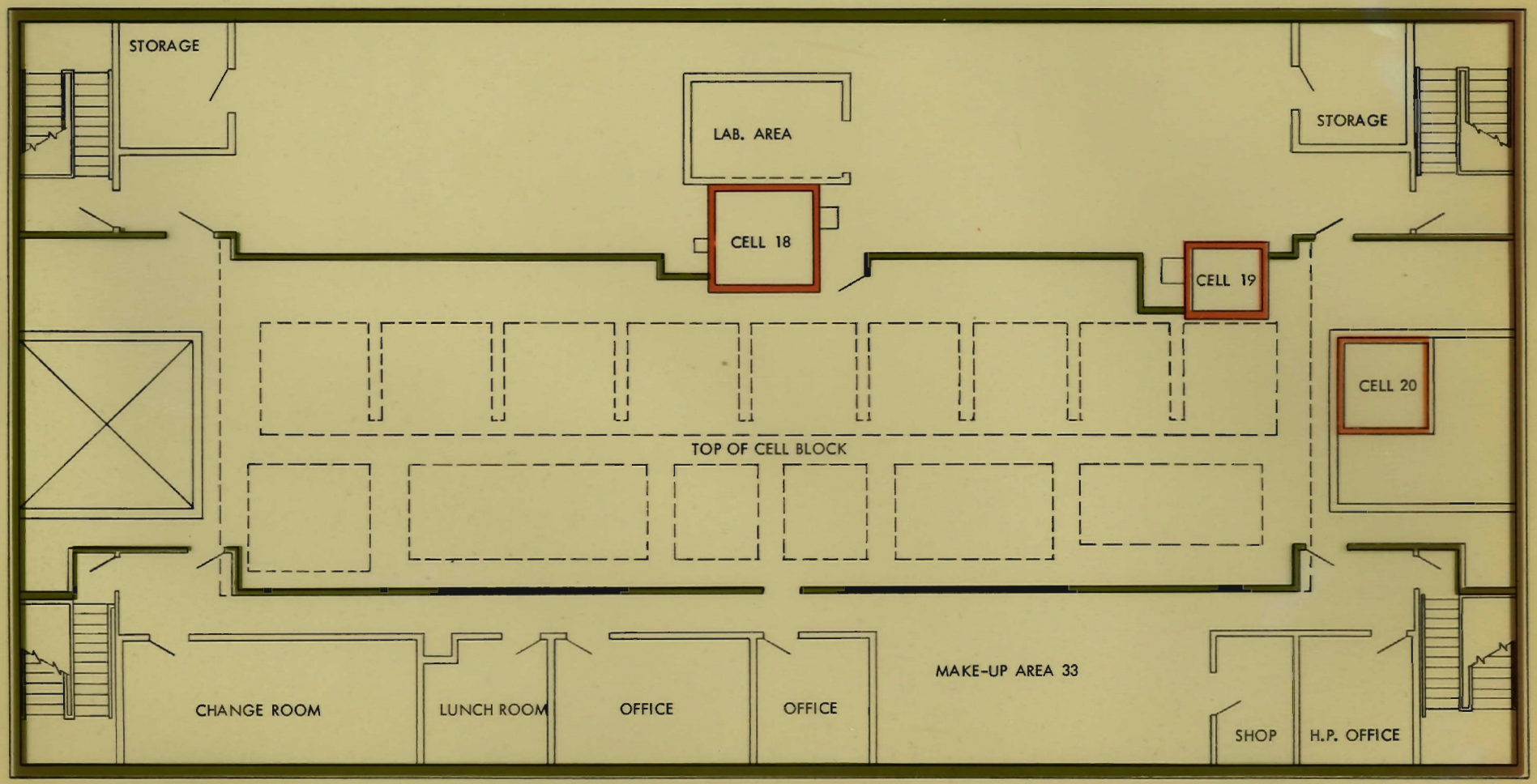

FIG. 3. BLDG. 3517 SECOND FLOOR PLAN

LIMITS OF SECONDARY CONTAINMENT

LIMITS OF PRIMARY CONTAINMENT

1.5 Process Description

The principal process equipment pieces are used in various sequences depending on the feed material and the product emphasized. The aqueous feed solution containing mixed fission products is concentrated by evaporation, and the fission products are then separated into groups of chemically related elements by inorganic precipitation or co-crystallization reactions. The concentrated fractions are processed by liquid extraction and by further precipitation and crystallization to obtain separated fission products of high purity. These purified fission product compounds are converted to a dried powder, which is pressed into pellets for insertion into containers. The containers are sealed by welding and shipped to the customer for use as a source of radiation or heat.

1.6 Criticality

Criticality is a consideration in Bldg. 3517 only in case of equipment failure coupled with an erroneous analytical result during the processing of a batch containing ~1000 g of Pu^{239} in Bldg. 3019. Any critical incident would occur behind 4 ft of concrete shielding.

1.7 Liquid Waste Systems

Liquid radioactive waste is collected in tanks in the F3P tank farm, where it is sampled for radiochemical analysis before it is jetted to tank W-5 in the tank farm. The process cooling water and the process steam condensate are collected in closed recirculation loops having surface heat exchangers for removing process heat. The excess water added to the system by condensate overflows into a collection tank, where it is sampled and analyzed for radioactivity before it is jetted to the process waste system. The operating area floor and sink drains, building steam condensate, and heat exchanger water drain by gravity to the process waste system.

1.8 Gaseous Waste Systems

Building 3517 has three gas handling systems, for (1) radioactive off-gas, (2) cell ventilation air, and (3) building ventilation air.

The radioactive off-gas system handles the gases and vapors from each process vessel and maintains the vessels at a negative pressure with respect to the cells. The individual lines are manifolded together to form a single 6-in.-dia line which discharges through a 4 N NaOH scrubber and a 6 N HNO_3 scrubber. The gases then pass through a heater, a stainless steel roughing filter, and an absolute filter and leave the building in a 6-in.-dia line going to the 3039 stack. The capacity of the system is 500 scfm with a normal throughput of 400 scfm.

The cell ventilation system maintains the service tunnel and cells at a negative pressure with respect to the remaining building. Individual ducts from each cell combine to form two 30-in.-dia ducts, which run out of the building to an underground filter pit containing roughing filters followed by absolute filters. The gases leave the filter pit through a single 48-in.-dia underground

duct and go to the 3039 stack. The capacity of the system is 12,000 scfm, although normal throughput is about 8000 scfm.

The building ventilation system provides air for personnel and the air to the cell ventilation system. Air is pulled into the building through roughing filters and absolute filters by a 12,000-scfm fan. The quantity of air is regulated to equal the air being removed by the cell ventilation system. If the building air becomes contaminated, a supplementary inlet connecting the building to the cell ventilation system opens automatically and rapidly exhausts the building air to the 3039 stack. This creates a negative pressure inside the building, which further ensures that there will be no diffusion of activity out of the building to the immediate environment. Figures 4 and 5 show the paths of building air during normal and emergency (air-borne activity) conditions.

1.9 Monitoring System

The continuous air monitors and radiation monitors in Bldg. 3517 are indicated in Figs. 4 and 5. A continuous activity sampler is located in the cell ventilation duct past the filter pit. The pressures of the vessel off-gas system and of the cells are indicated on gages in the operating area.

2.0 SUMMARY

2.1 Radioactive Material Content of Facility

Material	Design Capacity curies/year	Maximum Inventory in Plant, curies	Maximum Inventory in One Vessel, curies
Total fission products	1.6×10^6	1.2×10^6	5×10^5
Ce^{144}	1.2×10^6	1.0×10^6	5×10^5
Sr^{90}	6.0×10^4	3.0×10^4	3×10^4
Pm^{147}	3.0×10^5	1.0×10^5	1×10^5
Cs^{137}	2.0×10^5	9.0×10^4	4×10^4
Ru^{106}	1.0×10^5	3.0×10^4	3×10^4
Zr^{95}-Nb^{95}*	-	3.0×10^6	3×10^6
Heavy Elements**			
Pu^{239}	-	(30 g)	(30 g)
U^{235}	-	(3 g)	(3 g)
U^{238}	-	(750 g)	(750 g)
Am^{241}	-	30	30

*The Zr^{95}-Nb^{95} would occur only during processing of short-decayed (six months) fuel elements in Bldg. 3019 and would be held in an F3P tank for further decay before discharge to the ORNL waste system. Previous experience indicates that the major portion of the estimated 3.0×10^6 curies is removed during processing at Bldg. 3019 and does not appear in the feed to the F3P.

**The heavy elements are present as contaminants in the metal recovery waste stream. The maximum amount listed is that found in the most concentrated solution.

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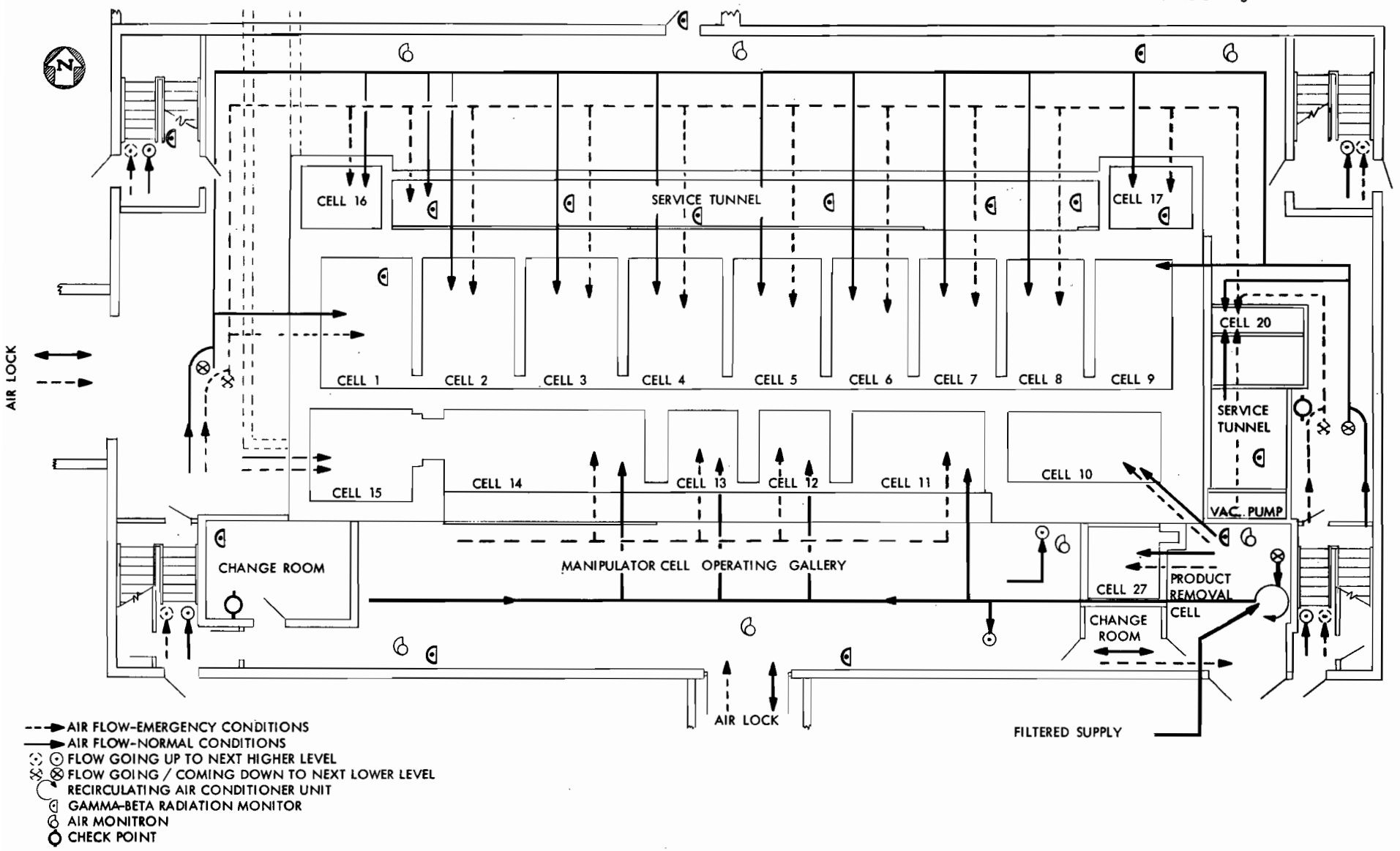

AIR FLOW-EMERGENCY CONDITIONS
AIR FLOW-NORMAL CONDITIONS
FLOW GOING UP TO NEXT HIGHER LEVEL
FLOW GOING / COMING DOWN TO NEXT LOWER LEVEL
RECIRCULATING AIR CONDITIONER UNIT
GAMMA-BETA RADIATION MONITOR
AIR MONITRON
CHECK POINT

FIG. 4. BLDG. 3517 - AIR FLOW - FIRST FLOOR PLAN

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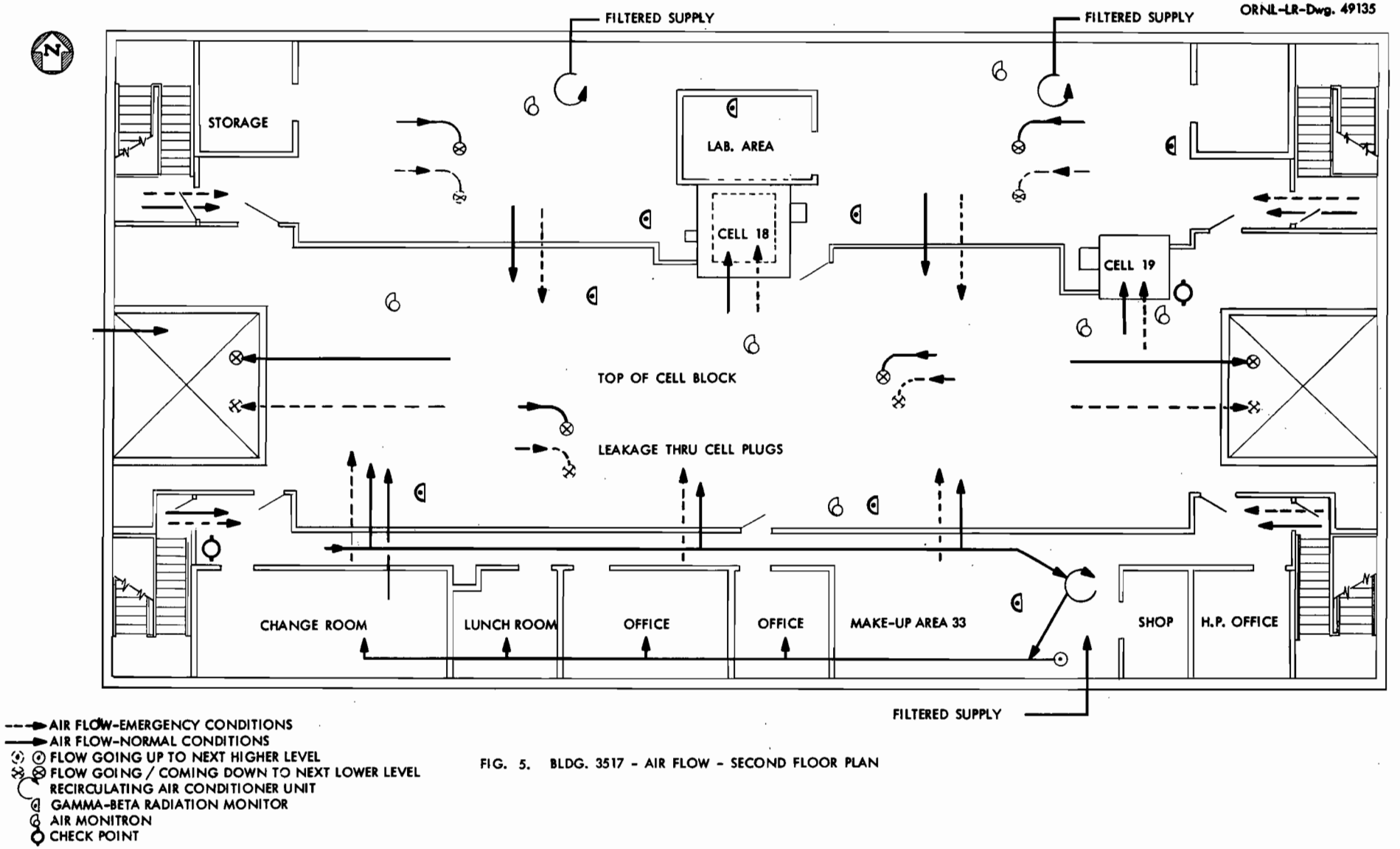

FIG. 5. BLDG. 3517 - AIR FLOW - SECOND FLOOR PLAN

2.2 Criticality Incident Potential

A criticality incident has been considered in which a sufficient quantity of fissionable materials (U^{235} or Pu^{239}) would accumulate with a rare earth fraction containing 100,000 curies of Ce^{144}, giving a single neutron burst resulting in 10^{18} fissions. The result of such a burst is the same as that shown in Sect. 2.4 for the Ce^{144} explosion.

2.3 Explosion and Fire Potential

The combustible or explosive materials used in the F3P are TBP, Amsco, ethyl hexanol, hydrazine, ammonia, and hydrogen. Possible reactions are ignition of a mixture of Amsco, HDEHP, and ethyl hexanol, nitration of TBP or a degradation product of TBP followed by heating beyond the decomposition point of the nitrated compound, reaction of 42.5% hydrazine with 12 N HNO_3 to form hydrazine nitrate followed by drying and heating of the product beyond its decomposition point, or combustion of ammonia or hydrogen in air. The flash points of the organics are:

Amsco-128	132°F (closed cup)
Ethyl hexanol	185°F (open cup)
TBP	295°F (open cup)

The maximum inventory of organics in the plant will be 450 lb of 0.4 M bis(2-ethylhexyl)orthophosphoric acid, 2.5 M ethyl hexanol in Amsco; in a single vessel, 150 lb of the same mixture. The most volatile component, Amsco, has a potential energy release of 19,000 Btu/lb. Preventive measures against fire or explosion in cells containing organics include an organic vapor detection system, the use of explosion-proof equipment, and the indication of temperature in the cells with alarm points. An automatic water spray system is actuated by rate-of-temperature-rise instruments in the cell containing organics.

Hydrogen occurs as a result of radiolytic decomposition of aqueous mixtures and is diluted well below its lower explosive limit of 4% by the vessel off-gas. Ammonia gas is sparged into acid solutions at a flow rate of 0.5 cfm, where approximately 95% of the gas is absorbed. Even if the entire flow should not be absorbed, the normal vessel off-gas would dilute the ammonia concentration to below the lower explosive limit of 16%.

The hydrazine--nitric acid reaction would involve the accidental addition of 12 liters of 42.5% hydrazine to 36 liters of 12 N HNO_3 to form 26,600 g of hydrazine nitrate. The potential energy release from hydrazine nitrate is 2,000 cal/g by thermodynamic calculations, but no information on the actual energy release or the conditions necessary for this release is available at this time.

2.4 Evaluation of Noncriticality Event Leading to Release of Radioactive Material

The distribution of radioactivities for noncriticality events is given in the following table:

	Sr^{90}	Ce^{144}	Ru^{106}
Total amount vaporized or suspended, curies	5.6×10^{-2}	7.7×10^{-1}	3×10^{4}*
Concentration, curies/m^3	1.32×10^{-3}	2.0×10^{-2}	-
Total amount released through VOG system, curies	-	4.8×10^{-3}	3.0
Max dose from VOG aerosol release, rem	-	1.3×10^{-5}	8×10^{-3}
Max dose from VOG gaseous fp's from criticality incident, rem	-	1.5×10^{-1}	-
Distance of dose downwind from stack, m	-	1760	1760
Total amount released through COG system, curies	7.8×10^{-4}	1.1×10^{-2}	-
Max dose from COG release, rem	4.2×10^{-5}	2.9×10^{-5}	-
Max dose from COG gaseous fp's from criticality incident, rem	-	1.6×10^{-1}	-
Distance of dose downwind from stack, m	1760	1760	-
Total amount released into bldg. or secondary containment, curies	2.4×10^{-5}	3.5×10^{-4}	-
Concentration, curies/m^3	2.4×10^{-9}	3.5×10^{-8}	-
2-min dose to bldg. personnel from aerosol release, rem	1.0×10^{-3}	7.0×10^{-4}	-
2-min dose to bldg. personnel from gaseous fp's in criticality release, rem	-	7.5×10^{-1}	-
Total amount leaking from bldg., curies (assuming ventilation system works)	3.82×10^{-7}	5.6×10^{-6}	-
Max downwind dose, rem	4.4×10^{-8}	3.3×10^{-8}	-
Distance for max dose, m	150	150	-
Fallout at 20 m downwind, curies/m^2	1.45×10^{-12}	2.1×10^{-11}	-
Total amount leaking from bldg., curies (assuming ventilation system fails)	8.65×10^{-6}	1.3×10^{-4}	-
Max downwind dose, rem	5.0×10^{-6}	3.6×10^{-6}	-
Distance for max dose, m	150	150	-
Fallout at 20 m downwind, curies/m^2	3.3×10^{-11}	5.0×10^{-10}	-
Total amount released** through stack, curies	7.8×10^{-4}	1.6×10^{-2}	3.0**
Fallout at 1500 m downwind, curies/m^2	2.0×10^{-13}	4.1×10^{-12}	negligible

*Total ruthenium released from centrifuge.

**Ruthenium leaves as RuO_4 gas.

Note: Gaseous fp's from the nuclear incident would result in less than 1 mr radiation outside the building.

2.5 Maximum Credible Accidents

The maximum credible accidents would be the explosive release of 100,000 curies of Ce^{144} from a centrifuge bowl, the explosive release of 30,000 curies of Sr^{90} from an evaporator, or a criticality incident in a centrifuge bowl or a mixer-settler tank containing 100,000 curies of Ce^{144}. The consequences of these accidents are described in Sect. 2.4.

3.0 PHYSICAL PLANT DESCRIPTION

Building Description

The Fission Products Pilot Plant (F3P) is located on White Oak Avenue between Third and Fourth Streets and is bounded on the northwest by Building 3505 (Metal Recovery); on the south by the waste disposal equalization pond; and on the east by Building 3508 (Transuranium Laboratory).(1)

The building structure(2) is two-story, standard concrete-block siding with an aluminum sided crane bay forming the third level. The building encloses 10,000 square feet of floor space and contains a total of 350,000 cubic feet of free space.

Cell Descriptions

The cells(3) at the F3P are divided into several categories and their characteristics are tabulated below.

Cell No.	Liner	Equivalent Standard Concrete Shielding (minimum)	Maximum External Radiation	Process
Process Type Cell				
1	S.S.	3 ft.	< 1 mr/hr	None
2	S.S.	4.5 ft.	< 1 mr/hr	F.P. precipitation
3	S.S.	4.5 ft.	< 1 mr/hr	F.P. precipitation
4	S.S.	4.5 ft.	< 1 mr/hr	F.P. precipitation
5	S.S.	4.5 ft.	< 1 mr/hr	F.P. precipitation
6	S.S.	4.5 ft.	< 1 mr/hr	F.P. evaporation
7	S.S.	4 ft.	< 1 mr/hr	Cs^{137} crystallization
8	S.S.	4 ft.	< 1 mr/hr	Cs^{137} crystallization
9	S.S.	4.5 ft.	< 1 mr/hr	R.E. solvent extraction
10	S.S.	4 ft.	< 1 mr/hr	R.E. mixer-settler
20	Amercoat	1 ft.	< 1 mr/hr	Off-gas scrubber
27	Amercoat	0.5 ft.	< 1 mr/hr	Off-gas scrubber

(continued)

(1) Appendix No. 1

(2) Appendix No. 2

(3) Appendix No. 3

Cell No.	Liner	Equivalent Standard Concrete Shielding (minimum)	Maximum External Radiation	Process
(continued)				
Manipulator Type Cell				
11	S.S.	4 ft.	< 5 mr/hr	Ce^{144} purification
12	S.S.	4 ft.	< 5 mr/hr	Ce^{144} pelletizing
13	S.S.	4 ft.	< 1 mr/hr	Cs^{137} purification
14	S.S.	4.5 ft.	< 1 mr/hr	Pellet storage and assembly
18	S.S.	1.5 ft.	< 1 mr/hr	Process control
19	S.S.	2 ft.	< 1 mr/hr	Process control
Loading Type Cell				
15	S.S.	2 ft.	< 1 mr/hr	Feed and product loading
Decontamination Type Cell				
16	Amercoat	1 ft.	< 5 mr/hr	Small equipment decontamination
Service Type Cell				
17	Amercoat	1 ft.	< 1 mr/hr	Cell 9 services
-	Amercoat	1 ft.	< 1 mr/hr	Service to cell
Tank Farm Type Cell				
21	Amercoat	4 ft.	< 1 mr/hr	F.P. concentrate storage
22	Amercoat	4 ft.	< 1 mr/hr	F.P. feed storage
23	Amercoat	2 ft.	< 1 mr/hr	Waste storage
24	Amercoat	2.5 ft.	< 1 mr/hr	Waste storage
25	Amercoat	2.5 ft.	< 1 mr/hr	Hot pipe tunnel
26	Amercoat	2.5 ft.	< 1 mr/hr	Hot pipe tunnel
Filter Pit Type Cell				
-	Amercoat	1.5 ft.	< 10 mr/hr	Cell ventilation filters

Six cells have personnel access doors which are entered via the cell door interlock system described under the section, Personnel Protection. All other cells are entered through sealed blocks or manholes. Of the six access doors, only those located on process control cells 18 and 19 enter into normal high-level radiation fields. These doors are locked and sealed as soon as operations commence and are not entered until decontamination proceedings have started. The entry to these cells is controlled by the Procedure for Personnel Entry to F3P Cells. (4)

(4) Appendix No. 7

The services to the various cells are tabulated below.

Cell Number	Steam 30 and 65 psi	Process Water	Chilled Water	Distilled Water	NH_3 25 psi	Electrical 440 V AC	Electrical 30 V DC	Electrical 110 V AC
1	X							
2,3,4,5	X	X	X		X	X	X	
6	X	X						
7,8	X	X	X					X
9	X	X			X	X	X	
10	X	X						
11	X	X	X	X	X	X		X
12,13,14	X	X	X	X				X
21,22	X	X						
23,24	X	X				X		

The plant is supplied with natural gas, but this supply has been blanked off outside the building since there has been no need for this type of service.

Process Equipment

The F3P cell equipment is divided into several categories and is tabulated in the following table.

Equipment Number*	Type Material	Volume Gallons	Agitation	Process
P-12	304 L S.S.	250	Agitator	Precipitation
P-23	304 L S.S.	250	Agitator	Precipitation
P-34	304 L S.S.	250	Agitator	Precipitation
P-45	304 L S.S.	470	Agitator	Precipitation
P-89	347 S.S.	50	Agitator	Pm^{147} Precipitation
P-511	347 S.S.	32	Agitator	Ce^{144} Precipitation
G-12	304 L S.S.	13	None	Centrifugation
G-23	304 L S.S.	13	None	Centrifugation
G-34	304 L S.S.	13	None	Centrifugation
G-45	304 L S.S.	13	None	Centrifugation
H-12	304 L S.S.	275	None	Centrifugate Hold-up
H-23	304 L S.S.	275	None	Centrifugate Hold-up
H-34	304 L S.S.	275	None	Centrifugate Hold-up
H-45	304 L S.S.	275	None	Centrifugate Hold-up
H-89	347 S.S.	90	None	Supernate Hold-up

(continued)

*First digit after letter is unit serial number in a particular category and last one or two designate cell where unit is located.

Equipment Number	Type Material	Volume Gallons	Agitation	Process
(continued)				
H-511	347 S.S.	110	None	Supernate Hold-up
F-12	304 L S.S.	280	None	F.P. Concentration
F-23	304 L S.S.	280	None	F.P. Concentration
F-34	304 L S.S.	280	None	F.P. Concentration
F-421	304 L S.S.	500	None	F.P. Concentration
F-521	304 L S.S.	500	None	F.P. Concentration
F-621	304 L S.S.	250	None	F.P. Concentration
F-821	304 L S.S.	250	None	F.P. Concentration
F-1021	304 L S.S.	250	None	F.P. Concentration
F-1121	304 L S.S.	250	None	F.P. Concentration
C-17	316 ELC S.S.	450	Agitator	Cs^{137} Crystallization
C-27	316 ELC S.S.	450	Agitator	Cs^{137} Crystallization
C-38	316 ELC S.S.	125	Agitator	Cs^{137} Crystallization
C-48	316 ELC S.S.	125	Agitator	Cs^{137} Crystallization
C-513	Glass	12	Agitator	Cs^{137} Crystallization
C-613	Glass	12	Agitator	Cs^{137} Crystallization
E-16	304 L S.S.	690	None	Evaporation
E-213	Tantalum-lined S.S.	5	None	Cs^{137} Product Evaporation
N-19	347 S.S.	55	Agitator	Solvent Contactor
N-29	347 S.S.	54	Agitator	Solvent Contactor
R-29	347 S.S.	90	None	Waste Receiver
R-39	347 S.S.	144	None	Waste Receiver
R-111	347 S.S.	42	None	Waste Receiver
S-122	304 L S.S.	5500	None	Feed Storage
S-223	304 L S.S.	3000	Agitator	Waste Storage
S-523	304 L S.S.	1000	Agitator	Waste Transfer
S-324	304 L S.S.	1000	Agitator	Waste Transfer
S-424	Glass-lined	500	None	HCl Waste Storage
P-110	347 S.S.	32	Agitator	Sr Precipitation
R-110	347 S.S.	165	None	Aqueous Waste Hold Tank
R-210	347 S.S.	165	None	Organic Extraction Hold Tank
R-310	347 S.S.	12	None	Organic Waste Hold Tank
R-410	347 S.S.	12	None	Organic Metering Tank
R-510	347 S.S.	45	None	R.E. and Sr Feed Tank
T-110	347 S.S.	0.25	None	Primary Air Lift Head Tank
T-210	347 S.S.	0.25	None	Secondary Air Lift Head Tank
Q-110	347 S.S.	1	None	Feed Metering Burette
E-110	347 S.S.	23	None	Raffinate Evaporator
E-210	347 S.S.	23	None	Strip Aqueous Evaporator
D-110	347 S.S.	-	None	E-110 Condenser
D-210	347 S.S.	-	None	E-210 Condenser
X-110	347 S.S.	50	Agitator	Mixer-settler Extractor
X-210	347 S.S.	50	Agitator	Mixer-settler Extractor
Y-110	347 S.S.	20	Agitator	Mixer-settler Organic Stripper
W-110	347 S.S.	2	Agitator	Raffinate Washer
W-210	347 S.S.	2	Agitator	Strip Aqueous Washer
V-128	347 S.S.	-	None	Organic Extractant Pump
N-120	347 S.S.	25	None	Off-gas Caustic Scrubber
N-220	347 S.S.	25	None	Off-gas Acid Scrubber

For location of equipment see Appendix No. 3.

Auxiliary Equipment

The chilled water unit consists of a 58-ton compressor unit with a pumped chilled water recirculation system to those vessels requiring additional cooling. The compressor system is cooled by a water recirculation system through a forced air cooling tower.

The building vacuum system is supplied by a two-stage Hy-Tor vacuum pump which has a recirculating water cooling system that drains into the hot waste system. A caustic scrubber and surge tank are located inside the manipulator cells to prevent suck-out of hot solutions from the Cs^{137} purification equipment.

Two 2000-gallon capacity stainless steel tanks are buried approximately eight feet below the ground level north of the F3P tank farm cells. These tanks are used for feed storage from Building 3019 and are equipped with liquid level instruments, cell ventilation, and process water cooling coils. The tanks are located on a crushed rock bed that has a drainage system which flows into the process waste system.

Six 500-gallon stainless steel lined shielded transfer containers are used for shipment of liquid feed from ICPP and are also used for transferring low-level product solutions within the ORNL site. The waste solution from ICPP has an average age of approximately four years. These tanks are equipped with valved Snaptite connections and electrical probes, and weigh approximately 20 tons. The maximum allowable external radiation is 200 mr/hr at contact. The normal shipments from ICPP have read 40 mr/hr at contact.

An open-air solvent storage shed is located approximately 60 feet from the northeast corner of the F3P building. The normal storage capacity is ten 55-gallon drums of di(2-ethyl hexyl) phosphoric acid and three 55-gallon drums of Amsco. All solvent is transferred in safety cans directly to the process solution addition funnel. No solvent is stored in the F3P building.

A caustic make-up and nitric storage area is located in an open area on the north side of the building. This area is used for transferring and storage of 400 gallons of 60% nitric acid and the make-up and storage of 1500 gallons of 19 M caustic. The acid and caustic are pumped through separate systems to the second level storage tanks for process addition.

Three chemical make-up and storage areas are located on the second level of the F3P building and all process chemicals are added to the cell equipment from these tanks. No make-up or storage tank is connected directly to a process solution addition line as each tank drains through a separate hose system. The total maximum storage capacity is 210 gallons of 60% nitric acid, 55 gallons of fuming nitric acid, and 400 gallons of 19 M caustic.

A nitrogen gas storage system is installed outside the northwest corner of the building. The capacity of this system is 12,000 cubic feet at 1000 psi and will be used for forming inert blankets during fission product source fabrication operations needing this protection. The

nitrogen supply is piped directly to Cell 14 and is connected remotely to the equipment.

All hot process solutions are transferred by steam jets or air lifts with the exception of the solvent waste pump located in Cell 10. A Lapp pulsafeeder pump can be used to pump feed from the storage tanks to E-16 evaporator, but the normal operation is by steam jet.

A bank of six 6-volt standard wet cell batteries is located on the outside north wall of the service tunnel to supply an emergency source of D.C. voltage to the braking circuit of the centrifuges in case of a power failure. An automatic charging system keeps the batteries at full charge.

4.0 CONTAINMENT

Cell Containment

The containment of radioactivity in the F3P cells is accomplished by sealing all roof plugs with oakum and G-K compound and sealing the access doors with a sealing compound. All access plug holes are welded on the inside of the cells and the plug is locked in position on the outside face of the cells to prevent their blowing out in the event of an explosion. The cell ventilation air intake ducts are filtered by glass wool roughing filters and have a check valve installed to prevent a blowback of activity.

In those cells requiring manipulator operations, the arms are sealed on the inside of the cell with a vinyl impregnated nylon bagging which protects the arm from contamination and effectively seals the manipulator arm opening into the cell. The master end of the arm is sealed on the outside of the cell with a similar bagging device which makes a double seal on the manipulator opening into a cell.

All spare lines into the cells are capped on the inside and the outside of the cell walls.

Building Containment

The F3P structure satisfies the requirements for containment of a radioactive release as specified by the Radioactive Operations Review Committee.

The structure is provided with air locks to all entrances of the building. The personnel access doors are air locked inside the building by enclosing the stairwells and requiring a separate entrance to the first and second floor operating area.

The loading areas are air locked on the outside of the building and are of sufficient size to allow the largest piece of equipment used to enter the structure. The west air lock permits a 44 foot tractor-trailer to enter the building to unload carriers and equipment. The doors are operated by a controller system which allows only one entrance door to be open at a time. The south air lock permits manipulators, fork lifts, and other small equipment to enter the manipulator cell area and is operated on the same principal as the west air lock. An air lock has been constructed over the north personnel entrance and encloses the tank farm sampling station.

All doors are gasketed and the air locks as well as the building structure have been treated with polyvinyl chloride and silicon membrane to minimize air leakage into the building.

Interior partitions are provided to enclose all operating areas, and mechanical cooling is installed for personnel comfort. The crane bay area which houses the top of the cells is partitioned from the operating areas, and is ventilated by a filtered air supply system.

All air is exhausted from the building by the cell ventilation system during normal and emergency conditions. The building air supply is limited to the cell ventilation exhaust capacity. The building air supply to the three normally occupied areas enters through a pre-filter and absolute filter bank located on the south side of the west air lock and furnishes the make-up air to the packaged type air conditioners. The supply air then passes from these areas into the cells and out through the cell ventilation exhaust system.

In the event of an accidental release of activity into the building interior from the cells, all building openings are closed automatically. The exhaust duct in Cell 20, which is partially opened at all times, is thrown to the full-open position and the interior of the building is exhausted to a vacuum of 0.5 inch W.G. The signal which actuates the automatic devices comes either from pressure sensors inside the cells or from a radiation monitor in the building.

The exhaust duct in Cell 20 is equipped with pneumatically regulated dampers which will maintain the building vacuum at least 0.5 inch W.G. above the vacuum in the cells. Building collapse by an excessive vacuum is prevented by a gravity balanced damper which is set to admit filtered air into the contained building space before the danger point is reached.

The make-up area tanks are exhausted by the hot off-gas system and the fumes from this source are scrubbed by the off-gas scrubber system.

5.0 CHEMICAL PROCESSES

The Fission Products Pilot Plant has operated on three general types of fission product waste solutions resulting from uranium fuel elements reprocessing. Two of these types resulted from ORNL (Building 3019 reprocessing of a special shipment of SRO fuel (Purex) and the reprocessing of BNL fuel. The third type (Redox) was transported from the Idaho Chemical Processing Plant.

The F3P operations satisfy two requirements of the Commission (1) to produce multikilocurie quantities of the long-lived fission products for distribution to private and public institutions and (2) to develop and test safe and economical methods for separating the fission products on a large scale.

The chemical processes which have been used in F3P to date are summarized in Appendix 4 and the operating procedures are included in Appendix 5. This composite flow diagram is intended to set forth the types of chemical operations which are performed but not necessarily in the order shown. The reasons for deviation from a process sequence are as follows:

1. A limited objective may be dictated by a special request from the AEC in which only one or possibly two isotopes are recovered.

2. The composition of the fuel reprocessing wastes may vary widely depending on the elements used to fabricate the fuel elements or the chemical process used for the U-Pu recovery. It is even possible that an F3P process will require considerable modification if the concentration of the gross contaminants (Fe, Al, SiO_2, etc.) should vary widely from batch to batch of the same fuel reprocessing sequence.

3. The development of new or modified processes in laboratory experiments must be transferred to F3P-level operations for final evaluation.

Chemical Reactions

The following sections are intended to describe the process chemistry without comment on the possible hazards involved in the operations. The operating procedures in Appendix 5 include the quantities of chemicals used, method of addition, and temperature of the solution. The chemical operations are numbered to correspond with the same operation shown in Appendix 4.

1. Zirconium-Niobium Precipitation Procedure (Appendix 5-e)

   The activities Zr^{95}-Nb^{95} are removed by adsorption on silica and separated from the solution by centrifugation. If there is insufficient SiO_2 in the waste solution, a slurry can be added.

$$SiO_2 + Zr^{95}\text{-}Nb^{95} \longrightarrow SiO_2\text{-}Zr^{95}\text{-}Nb^{95} \text{ (adsorbed ions)}$$

1-A. SiO_2-Zr^{95}-Nb^{95} Cake Dissolution (Appendix 5-e)

The SiO_2 cake is digested with hot NaOH and the residue slurried to the waste system.

$$2\ NaOH + SiO_2 \longrightarrow Na_2O \times SiO_2$$

2. Iron-Ruthenium Precipitation Procedure (Appendix 5-f)

If the solution does not contain sufficient iron, $Fe(NO_3)_3$ is added.

$$Fe(NO_3)_3 + Ru(NO_3)_3 + NH_3 + H_2O \longrightarrow Fe(OH)_3 + Ru(OH)_3 + NH_4NO_3$$

3. Rare Earth Precipitation Procedure (Appendix 5-g)

$$R.E.\ (NO_3)_3 + 3\ NH_3 + H_2O \longrightarrow R.E.\ (OH_3) + 3\ NH_4NO_3$$

3-A. R.E. $(OH)_3$ Cake Dissolution in HNO_3 (Appendix 5-g)

$$R.E.\ (OH)_3 + 3\ HNO_3 \longrightarrow R.E.\ (NO_3)_3 + 3\ H_2O$$

3-B. Oxalate Precipitation Procedure (Appendix 5-j)

$$2\ R.E.\ (NO_3)_3 + 3H_2C_2O_4 \longrightarrow R.E._2(C_2O_4)_3 + 6\ HNO_3$$

$$\text{Side reaction } C_2O_4^- + \text{radiation} \longrightarrow 2\ CO_2$$

3-C. R.E. (C_2O_4) Dissolution in HNO_3 (Appendix 5-j)

$$R.E._2(C_2O_4)_3 + HNO_3 + Mn^{++}\ (\text{catalyst}) \longrightarrow$$

$$R.E.\ (NO_3)_3 + 6\ CO_2 + NO_2$$

$$\text{Side reaction } C_2O_4^- + \text{radiation} \longrightarrow 2\ CO_2$$

3-D. Yttrium Extraction into D2EHPA (Appendix 5-k)

Yttrium is extracted into Di(2-ethyl hexyl) phosphoric acid with Amsco as an organic diluent from a 1 N HNO_3 solution.

$$Y(NO_3)_3 + 6D2EHPA \longrightarrow Y(D2EHPA)_6 + 3\ HNO_3$$

3-E. Cerium Oxidation and Extraction (Appendix 5-l)

To a 1 N HNO_3 solution containing Ce, $KMnO_4$ is added to obtain Ce^{+4} which is then extracted into D2EHPA-Amsco.

$$KMnO_4 + 3Ce(NO_3)_3 + 4\ HNO_3 \longrightarrow$$

$$MnO_2 + 3\ Ce(NO_3)_4 + KNO_3 + 2\ H_2O$$

$$Ce(NO_3)_4 + xD2EHPA \longrightarrow Ce(D2EHPA)_x + 4\ HNO_3$$

3-F. Cerium Recovery from Organic Solution (Appendix 5-1)

$$2Ce^{+4}\ (\text{organic phase}) + \overset{(2N)}{HNO_3} + H_2O_2 \longrightarrow 2\ Ce(NO_3)_3 + O_2$$

3-G. Cerium Oxalate Precipitation (Appendix 5-o)

$$2Ce(NO_3)_3 + 3\ H_2C_2O_4 \longrightarrow Ce_2(C_2O_4)_3 + 6HNO_3$$

$$\text{Side reaction } C_2O_4^- + \text{radiation} \longrightarrow 2\ CO_2$$

3-H. Calcination of $Ce_2(C_2O_4)_3$

$$Ce_2(C_2O_4)_3 + \text{heat} \xrightarrow{\text{air}} 2\ CeO_2 + 6\ CO_2$$

3-I. Promethium Oxalate Precipitation (Appendix 5-m)

Chemistry same as 3-G.

$$\text{Side reaction Citric acid} + HNO_3 + MnO_2 \longrightarrow CO_2 + (CH_3)_2CO + Mn$$

(citric acid)

3-J. Promethium Oxalate Dissolution (Appendix 5-m)

Chemistry same as 3-C.

3-K. Promethium Purification Procedure

The Pm^{147} is separated from the remaining rare earth elements by continuous mixer-settler operations in a TBP-HNO_3 system. The chemical process description and procedures are described in Appendix 5-s.

4. Strontium Precipitation Procedure (Appendix 5-h)

The alkaline earths are precipitated as the carbonates

$$\begin{matrix}\text{Ca}\\ \text{Sr}\\ \text{Ba}\end{matrix}(NO_3)_2 + Na_2CO_3 \longrightarrow \begin{matrix}\text{Ca}\\ \text{SrCO}_3\\ \text{Ba}\end{matrix} + 2\ NaNO_3$$

4-A. Alkaline Earth Carbonate Dissolution (Appendix 5-h)

$$\text{A.E. } (CO_3) + HNO_3 \longrightarrow \text{A.E. } (NO_3)_2 + CO_2 + H_2O$$

4-B. Barium Separation

$$Ba(NO_3)_2 + Na_2CrO_4 \longrightarrow BaCrO_4 + 2\ NaNO_3$$

4-C. Dissolution of $BaCrO_4$

$$BaCrO_4 + HNO_3 \longrightarrow \text{soluble in acid solution}$$

4-D. Same as 4-A.

4-E. Calcium Separation from Strontium

The Sr^{90} is separated from the Ca by continuous mixer-settler operations in an acetic acid-HDEHP system. The chemical process description and procedures are described in Appendix no. 5-t.

5. Technetium Precipitation Procedure (Appendix 5-i)

Technetium will coprecipitate with a magnetic iron hydrated oxide precipitate believed to be $Fe_3O_4 \cdot x\ H_2O$. The $Fe_3O_4 \cdot x\ H_2O$ is formed from an acid solution by a partial reduction of Fe^{+3} to Fe^{+2} and then by making the solution basic with NaOH.

$$Fe^{+3} + N_2H_4 \longrightarrow Fe^{+2} + N_2 + NH_3$$

$$Fe^{+2} + Fe^{+3} + NaOH \longrightarrow Fe_3O_4 \cdot x\ H_2O$$

Side reaction $NH_4NO_3 + NaOH \longrightarrow NaNO_3 + NH_3 + H_2O$

5-A. $Fe_3O_4 \cdot x\ H_2O$ Dissolution

$$Fe_3O_4 \cdot x\ H_2O + 9HNO_3 \longrightarrow 3\ Fe(NO_3)_3$$

5-B. Iron Precipitation by NH_3 Gas

Technetium does not coprecipitate on a $Fe(OH)_3$ precipitate while most of the remaining fission product contaminants are scavenged from the solution.

$$Fe(NO_3)_3 + 3\ NH_3 + H_2O \longrightarrow Fe(OH)_3 + 3\ NH_4NO_3$$

5-C. $Fe(OH)_3$ Cake Dissolution

$$Fe(OH)_3 + 3HNO_3 \longrightarrow Fe(NO_3)_3 + 3H_2O$$

6. Crystallization Procedure (Appendix 5-a)

Cesium-137 is recovered from solution by cocrystallization with ammonium alum. The alum may be added as the compound or formed in the solution by supplying the missing ions. For example, the Redox solution contains Al and NH_4 ions as received which may be converted to alum by the addition of Na_2SO_4 to supply the sulfate ions to yield $(NH_4)_2\ Al_2(SO_4)_4$.

$$2NH_4NO_3 + 2Al(NO_3)_3 + 4Na_2SO_4 \longrightarrow (NH_4)_2\ Al_2(SO_4)_4 + 8NaNO_3$$

6-A. Cs_2PtCl_6 Precipitation (Appendix 5-p)

$$Cs_2Al_2(SO_4)_4 + H_2PtCl_6 \longrightarrow Cs_2PtCl_6 + Al_2(SO_4)_3 + H_2SO_4$$

6-B. Reduction of Platinum to the Metal (Appendix 5-p)

$$Cs_2PtCl_6 + N_2H_4 \longrightarrow 2\ CsCl + Pt + 4HCl + N_2$$

$$\text{Side reaction } 3N_2H_4 + Pt \longrightarrow 4\ NH_3 + N_2$$

6-C. Dissolution of Pt Metal (Appendix 5-p)

$$Pt + 6HCl + H_2O_2 \longrightarrow H_2PtCl_6 + H_2O + O_2$$

6-D. Oxidation of Ammonium Ion (Appendix 5-p)

$$2NH_4Cl + HNO_3 + HCl \longrightarrow N_2$$

7. Mercury Precipitation Procedure (Appendix 5-b)

$$Hg(NO_3)_2 + NaH_2PO_2 \longrightarrow Hg + NaH_2PO_4$$

7-A. Mercury Dissolution in HNO_3 (Appendix 5-b)

$$3\ Hg + 8HNO_3 \longrightarrow 3Hg(NO_3)_2 + 2NO + 4H_2O$$

7-B. Magnetite Precipitation (Appendix 5-c)

Same as No. 5.

7-C. Same as 5-C (Appendix 5-c)

Operating Safeguards

The process operations at F3P are accomplished by standard operating procedures(1) and any deletion, omission or addition to these procedures without approval of the building supervisor are considered as grounds for disciplinary action. The usual chemical plant safety practices are followed and specially applicable regulations are published(2).

The operating procedures are written in such a manner that the physical operation is always the same although the chemical reaction is different. For example, the magnetite precipitation operation is the same as the oxalate precipitation operation except for the chemical addition and the digestion periods. The crystallization procedures in six different crystallizers are identical except for quantities of chemicals involved.

The operators follow a standard operating procedure book located at the panelboard and record the critical data on a process data card(3) which is filed for each batch processed. The batch data card is used for

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(1) Appendix 5.

(2) Appendix 9.

(3) Appendix 15.

inventory control as well as a check system on the actual operations. These cards remain in the inventory control board until all analyses have been received and several batches have been combined for further purification. The completed cards are then filed for future reference.

Changes in operating procedures are continually taking place due to the varying conditions of the feed material. The changes are reviewed by the building supervisor and a chemist from the Radioisotope Development Department. If a procedure change is approved, the operating procedure at the panelboard is corrected and written notification is recorded by the building supervisor in the "Instructions to Foremen Log."

Decontamination operations vary depending on the equipment involved but a standard decontamination solution procedure(4) is used to prevent the faulty mixing of chemicals used during this period. The flow of the decontamination solutions is determined by the building supervisor and all jets and valves leading to critical areas are blocked and tagged at the panelboards.

Shift tank inventories(5) are taken at the end of each shift and the recorded data are studied by the incoming foreman to assure continuity of operations. Additional data are recorded in the Operations Log Book by each shift foreman in order to have a written record of all operations taking place at the F3P. The log books when filled are filed for future reference.

A building check sheet(6) is filled out once a day to record the data on the building services and auxiliary equipment. All stored chemicals used in the process are regularly inventoried.(7)

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(4) Appendix 10.

(5) Appendix 11.

(6) Appendix 12.

(7) Appendix 14.

6.0 PERSONNEL PROTECTION

Personnel Exposure

The period from January 1, 1959, to December 31, 1959, is representative of the typical radiation problems encountered at the F3P. This period covered normal operations on solutions containing from 5 curies/liter to 6500 curies/liter and included a decontamination of the plant facilities. However, this did not include decontamination of manipulator cells for Ce^{144} final purification. This period also includes exposure from emergency work performed for the Operations Division during the final quarter. The exposure from normal operations was less than 10 mr (d_p) per week per man. The highest individual exposure was 360 mr (d_p) per week which occurred during the decontamination of the cell ventilation filter pit. The following data were calculated from the Health Physics exposure data for 1959:

Personnel	Total Dosage/Year mr (d_p)	Total Weeks Worked	Average Weekly Dosage mr/wk (d_p)	Highest Weekly Average Dose mr/wk (d_p)	Lowest Weekly Average Dose mr/wk (d_p)
Operators	18,965	1,050	18.7	29.1	8.9
Supervision	6,020	364	16.5	25.7	9.8
Health Physics J.H. Pemberton	1,955	50	39.1	39.1	39.1

Exposure Possibilities

The largest source of exposure will come from decontamination work or accidental release of contamination. The decontamination work exposure is organized under standard procedures as listed in the Zoning Procedure for the Fission Products Pilot Plant(1) and the Procedure for Personnel Entry to F3P Cells.(2)

All samples and solid waste material are removed from the manipulator cells into a bottom loading carrier which has no greater reading than 200 mr/hr at contact. The normal exposure for this type of work would be less than 10 mr per week per man.

Entrance into hot cells by access door is controlled by the building foremen through the interlock system and has to be approved by a Health Physics representative. Entrance by way of the top of the cells is controlled by Health Physics through the Zoning Procedure for the Fission Product Pilot Plant(1) and Procedure for Personnel Entry to F3P Cells.(2)

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(1) Appendix 6.

(2) Appendix 7.

Radiation and Contamination Controls

Radiation Monitrons

Sixteen radiation monitors are located at strategic points in the building and six Victoreen monitors are located in the service tunnel for personnel protection during maintenance operations. The monitors in those areas considered most critical, in regard to a potential high radiation level, are tied to a central alarm system which is answered by supervision and Health Physics if the alarm signal is sounded. Monitors in areas of minor potential for radiation and contamination are on a local alarm system which is answered by supervision. All monitron units are checked for operation on a daily basis.

Constant Air Monitors

Four portable beta-gamma constant air monitrons with local alarms are located in the critical areas which could be affected by a high air count. One unit is always available for movement to take direct readings during decontamination and maintenance work. One air monitor station is located on the south wall of the manipulator cell area and is used to actuate the outer door interlock system. If a masking tolerance air count is recorded on this air monitor, it will ring a local alarm and lock all the outside entrance doors to prevent personnel from entering the building. The outer door lock system can also be actuated by supervision throwing a manual switch in the case where the building floors are contaminated. All doors can be unlocked by pushing the normal crash-bar hardware from inside. All constant air monitors are checked for operation daily and the outside entrance door interlock is checked on a weekly basis.

Radiation Detection Equipment

Victoreens and cutie-pies are located in strategic sections of the building and all operating personnel are trained in the use of these instruments.

A hand and foot counter is located adjacent to the locker room and all personnel are required to check themselves before entering the lunchroom or leaving the building area. A probe is also available for checking beta and gamma contamination on the outer garments.

A Health Physics office is manned on the day shift and contains an alpha smear counter, a beta-gamma smear counter, high-level radiation probes, portable disc air samplers, high vol. air samplers, plus additional cutie-pies and Victoreens. All operating personnel are trained in the use of this equipment.

Emergency Equipment

Emergency combat mask cabinets are located in the operating areas for immediate use in case of a high air count in the building. Individual gas masks with prescription safety glasses have been issued to

personnel who must have prescription glasses for vision while wearing the masks.

An emergency cabinet is located on the second level west and contains an air line mask system, plastic suits, rubber suits and chem-ox gas masks.

An emergency evacuation procedure(3) is posted in the building and all personnel have been informed of their duties and procedure.

Cell Door Interlock System

The service tunnels and Cell 15 loading station are protected by a cell door interlock system. The Cell 15 system consists of an electrical lock system on the cell door entrance, a pneumatic lock system on the shield between Cell 14 and Cell 15, and a manual bar lock on Cell 15 roof plug. A special padlock is used on Cell 15 door in case of electric failure on normal switches. The foremen have possession of the key for this lock. The system is designed in such a manner that it is impossible for personnel to be in Cell 15 without supervision present when the shield or the loading station door between Cell 14 and Cell 15 is raised. Cell 14 will be a source of very high level radiation. If Cell 14 shield is raised, the Cell 15 block can not be opened and Cell 15 personnel access door is electrically locked. To enter Cell 15 would require the shield and the loading station door to be lowered into place and the shift foreman and at least two other employees to open the personnel access door. When the shift foreman turns the spring loaded electrical lock with his key, the electrical lock is deenergized. The shift foreman can not leave this switch until two other employees open the door. As soon as the cell door is cracked open, a switch energizes a pneumatic locking device on the second level which bolts the shield in place and deenergizes the loading station crane. This shield and the loading station door can not be raised until the Cell 15 door is closed by supervision. The electrical switch box for this circuit is padlocked in the "on" position and the building supervisor and Lab Shift Supervisor have the master keys.

The service tunnel interlock system is electrically operated and is locked in the "on" position at all times. If maintenance is required, the shift foreman schedules a panelboard shutdown and then deenergizes the system by turning a spring loaded electrical switch with his key. The foreman can not leave the switch until one other employee opens the door to the service tunnel. When the door is open, instrument air to the panelboards is turned off by an electrical solenoid valve and no process operations can take place in the process cells. During the maintenance period, the employees are protected by six Victoreen monitron units and a standard monitron which rings a local alarm and a central alarm at the panelboards. No personnel may enter the service tunnel without a radiation survey by supervision or a Health Physics representative.

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(3) Appendix 8.

To close the service tunnel, supervision must walk from one end of the service tunnel to the other and operate two button switches within a three minute time limit in order to lock the service tunnel doors. As soon as the doors are locked, the instrument air is automatically turned on to the panelboards and operations can be started. The service tunnel doors are also padlocked to prevent entry during a power failure. The electrical circuit to the interlock system is padlocked and the building supervisor and the Lab Shift Supervisor have the master key.

Process Safeguards

All panelboard instrumentation is protected from radioactive back-ups by differential-pressure cells located in the service tunnel or in shielded cubicles. Any back-up of radioactive solution will be contained in these areas and will be handled under the normal decontamination procedures.

Solution addition to the various process vessels takes place in the operating areas through valved funnels. In the case of a vessel pressurizing during solution addition, a flapper-valved funnel in the process cell will open and dissipate the pressure into the cell. The flapper-valved funnel in the solution addition line is equivalent to an open solution addition funnel in the process cell but eliminates the loss of off-gas capacity to the tank.

The ammonia sparging system to the precipitator vessels is controlled by air-to-open valves and has a constant air purge system to prevent suck-back due to NH_3 gas dissolving in the solution and creating a vacuum in the line.

All F3P instrumentation is designed to fail closed in case of an emergency. The only exceptions to this rule is the off-gas header control valves and the evaporator (E-16) condenser water. These controls are instrumented to fail open under emergency conditions to prevent pressurization of the process system.

The radioactive process solution transferring by steam jets is protected against back-ups by a check valve-vent system. The vacuum formed by the condensation of steam in the line after the jet is closed is relieved by a check valve to a vent header which terminates inside the process cell. The operating experience with this system has indicated that it is a satisfactory automatic preventative of radioactive solution in steam jet lines.

The process vessels where precipitation, crystallization, evaporation, solvent extraction and high-level storage take place are protected by an alarm system which indicates high temperature, high level or low off gas. All alarms of this type are signaled at the panelboard and are answered by the operating personnel.

The solvent extraction equipment is protected against fire hazard by a temperature regulated fire water spray system. If the cell temperature rises above 50°C, the sprinkler system is automatically turned on to spray the cell equipment.

All cell plugs are sealed on the inside of the cell and bolted to the concrete on the outside of the cell to prevent blowing out in the case of an explosion in the cells.

The radioactive powder storage well in Cell 14 is cooled by a process water cooling coil in a bath of distilled water. A high and low liquid level probe system alarm has been installed to keep the bath at the proper operating level. A high temperature alarm has been installed to prevent the well from overheating. These alarms are answered by the operating personnel.(4)

The house vacuum system is protected by a surge pot located in Cell 11 and is equipped with a liquid level alarm system to prevent overflowing. The alarm is answered by the operating personnel.

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(4) Appendix 13.

WASTE DISPOSAL

Liquid Waste

Low Level Radioactive Waste

Low level radioactive wastes consist of cell floor washings, cell sumps solutions, cell decontamination solutions, service tunnel floor washings, and evaporator condensate. They are collected in two stainless steel waste collection tanks, S-223 and S-523. The normal volume of this type of waste is 1000 gallons per day and the activity level is 50 curies of mixed fission products per day. These solutions can be sampled, but under normal operation are jetted to W-5 tank on the ORNL tank farm when the tank liquid level reaches 60% of capacity. In the case of a tank bursting in the cells, the contents would drain to the low level collection system and would be contained in this manner until the building supervisor determined the best disposition of this solution.

Intermediate Level Radioactive Waste

The intermediate level radioactive waste solutions consist of the waste stream from the normal process operations plus decontamination solutions. These solutions are collected in a stainless steel tank, sampled for radiochemical analysis and then jetted to W-5 tank in the ORNL tank farm. The normal volume of these solutions is 2000 gallons per day and the average activity is 250 curies of mixed fission products per day. These solutions are generally basic, but could be acidic from nitric acid decontamination solutions.

A special boltaron waste system collects the intermediate level waste from the hydrochloric acid system used for Cs^{137} purification. The hydrochloric acid wastes are collected in a glass lined tank, sampled for radiochemical analysis, neutralized with sodium hydroxide and jetted to W-5 tank in the ORNL tank farm.

High Level Radioactive Waste

High level radioactive waste streams consist of short-lived radioisotopes such as Zr^{95}-Nb^{95} which are waste products from processes using short-cooled fuel elements. The normal flow is 150 gallons per day with an average activity level of 50,000 curies per day. Sixty percent of this activity is Zr^{95}-Nb^{95}.

These waste solutions are collected in a 5550-gallon stainless steel storage tank (S-122) in the F3P tank farm and are allowed to decay to an intermediate level waste. If process commitments do not allow a sufficient decay period, the solution is sampled for radiochemical analysis and jetted to W-6 tank in the ORNL tank farm for storage and further decay. This procedure is done only with divisional approval.

Organic Waste

The organic waste from F3P consists of Amsco, TBP and di(2-ethyl hexyl) phosphoric acid which have been stripped of practically all of the

activities which were extracted in the process. The volume of organic waste will normally be from 250 to 500 gallons per month with a possible maximum rate of 700 gallons per month. This waste is pumped directly to tank W-5 or W-6 in the ORNL tank farm system after it is stripped.

Process Waste System

The process waste system collects the building steam condensate, distilled water unit condenser water, vacuum pump cooling coil water, and the normal building sink and floor drains. The latter system has very small flow and the contamination in this system would originate from minor spills in the operating area. The vacuum pump cooling system is operated in a closed system with no possibility of contamination entering this stream.

The process cooling water system consists of two separate recirculation loops. One loop containing chilled water is used in the cooling coils in the crystallizer section. The process heat in the water is removed in a 58-ton chilled water refrigeration unit and recirculated to the cooling coils. In the other loop the process heat is removed in heat exchangers cooled by ORNL process water, which is discharged to waste. The F3P process water is recirculated to the vessel cooling coils. Condensate from process vessels is also collected in this system. The excess water due to the condensate overflows into a collection tank where it is sampled for an activity determination before it is jetted to the ORNL process waste system.

Storm Sewer System

The storm sewer system receives all roof drainage, building heating condensate, and cooling water to the freon condenser of the chilled water unit. All of these sources are non-radioactive and the only possibility of contamination would come from an outside source which would settle on the building roofs. The normal operation of the chilled water unit is to recirculate the cooling water from the compressor unit to a cooling tower, but during maintenance shutdowns on the cooling tower, the cooling water would go through the freon condenser system directly to the storm sewer. There is no possibility of contamination occurring in this system.

Gaseous Waste

Off-Gas System

The off-gas system at the F3P supplies negative pressure to every vessel in the plant cells. The negative pressure is controlled in Cell 20 by two pneumatically operated valves. One pneumatic valve automatically adjusts the off gas to 6 to 8 inches of negative water pressure on the process and tank farm cell vessels. The normal flow through this controller is 400 cfm and the gases present are NH_3, $Ru^{106}O_4$, and oxides of nitrogen. The particulate matter from entrainment could be any fission product radioisotope that would be in process. However, samples from the off-gas line show only $Ru^{106}O_2$ as the main offender to date. The other pneumatic valve maintains a negative water pressure of 12 to 14 inches on the manipulator cell equipment. The normal flow through this system is 200 cfm and the gases present are NH_3, $Ru^{106}O_4$, and oxides of nitrogen.

The particulate matter would be $Ce^{144}O_2$, $Cs^{137}Cl$, and $Sr^{90}TiO_3$. The additional off gas on the manipulator cells is needed to overcome the pressure drop of an additional scrubbing unit located in Cell 13 which consists of a glass bubble cap, continuous flow, caustic scrubber. This scrubber removes hydrochloric acid fumes from the Cs^{137} purification equipment to prevent corrosion in the main stainless steel off-gas line.

Both off-gas systems tie-in into a common header for scrubbing action in Cell 20. The off gas is scrubbed in a nitric acid countercurrent flow Berl saddle scrubber to absorb ammonia gas. From the acid scrubber, the off gas flows into a caustic countercurrent flow Berl saddle scrubber to remove gaseous $Ru^{106}O_4$ and oxides of nitrogen. The off-gas stream flows through an entrainment separator, heater, stainless steel "Neva-Clog" pre-filter, and an absolute filter to remove final particulate matter. From this point the off gas flows into the 3500 Area off-gas system. The off-gas scrubber cell is instrumented to record pressure drops and flow in cfm, and is equipped with a contamination detection system which is a part of the Operations Division monitoring system in the outlet stream. A dual filter system is used to continue filtration of the off gas while filter change-outs are taking place. The building off-gas system is designed to fail open in case of an emergency.

Cell Ventilation System

The cell ventilation system for the F3P area draws a vacuum of 0.5 inch to 1.0 inch W.G. on the process cell, manipulator cells, tank farm cells, service tunnels, decontamination cell, pipe tunnels, and W-19 and W-20 feed storage tanks. The normal volume of air swept through this system is 4000 cfm.

The filtration of the cell ventilation air consists of stainless steel "Neva-Clog" roughing filters, which can be washed by chemical sprays, and a bank of CWS filters for absolute filtration. These units are gasketed and sealed in place by a locking device to prevent leakage around the filters. A monitor station is located on the outlet duct approximately 20 feet from the filter pit and is used to monitor the activity and measure the actual flow. An automatic electric sump pump removes the water leakage into the filter pit and in case of pump failure, an automatic steam jet system goes into operation. All ground water leakage and decontamination solutions are piped to S-223 tank in the F3P tank farm system.

In addition to the building filtering system, the air entering the cells is filtered by glass wool roughing filters. Manipulator cell Nos. 11, 12, 13 and 14 have individual stainless steel roughing filters on the cell ventilation ducts, which remove the majority of the powdered $Cs^{137}Cl$, $Ce^{144}O_2$, and $Sr^{90}TiO_3$ produced and handled in these particular cells.

A small by-pass system with a capacity of 1000 cfm has been installed on the F3P cell ventilation filter pit and is equipped with CWS absolute filters. This is an operational by-pass to give a slight negative pressure on the F3P cells during the period needed for changing the filters in the main filter pit.

Solid Waste

High Level Solid Wastes

High level solid wastes from the F3P consist of materials and equipment used in the manipulator cells that become contaminated with mixed or separated fission products. Any material or equipment reading greater than 1 r/hr is washed in the manipulator cell, placed in a plastic bag, inserted in a one-gallon tin can, sealed with a lid and removed from the cell into a bottom-loading uranium-shielded carrier. The carrier is then removed to the burial ground by the riggers under the surveillance of a Health Physics representative. At the burial ground, the bottom drawer is opened remotely and the can is dropped into the hot waste trench. A bulldozer pushes enough dirt into the trench to give adequate shielding for future operations. The carrier is returned to the building, smeared and cleaned to the building tolerances of 500 d/m/100 cm^2.

A two-gallon capacity lead shielded carrier can be used in Cells 11, 12, 13, and 14 in conjunction with the Cell 15 loading station. This carrier has to be loaded from the top with the aid of manipulator arms and the handling procedure is identical to that described above. The normal reading at contact on the solid removal carriers is less than 100 mr/hr. Any reading greater than 100 mr/hr requires the material to re-enter the cell for further decontamination.

Large equipment such as laboratory balances, furnaces, presses, scales, stainless steel filters, lab centrifuges, etc., have to be decontaminated to less than 1 r/hr before they may be removed from a cell. If they are to be discarded, a lead-lined Dempster Dumpster is used to remove the material to the burial ground. If the equipment can be recovered by further decontamination, it is taken by the operating personnel to decontamination Cell 16 where it can be further decontaminated with low exposure to personnel.

Low Level Solid Wastes

The low level solid wastes consist of contaminated swabs, sanitary pads, mops, blotting paper, kraft paper, or other materials used in manual decontamination procedures. This material normally reads less than 100 mr/hr and is placed in plastic-lined hot cans located in strategic areas in the building. When a can is full or the radiation level of 6 mr/hr is exceeded, the plastic bag is sealed with tape, the lid placed on the can and the can removed to the hot-can storage shed. The hot-can truck removes the material to the burial ground on a daily basis. The returning empty hot cans are checked for contamination by Health Physics and cleaned to building tolerances before re-entering the building.

A daily survey of the hot cans is made by the Health Physics representative and supervision surveys suspected containers during shift decontamination operations.

PROCESS HAZARDS

Chemical Reactions

Gas Formation

The normal process operations at F3P include chemical additions or reactions which form various types and quantities of gases. These operations are tabulated below.

Process	Chemical Addition	Chemical Reaction	Gas Per Batch: Liberated	Gas Per Batch: Vol. (l)	Gas Per Batch: Est. Rate l/min
Magnetite Ppt.	Hydrazine	Fe^{+++} Reduction	N_2	935	31
Cs^{137} Purification	Hydrazine	Pt Reduction	N_2	200	7
Cs^{137} Purification	HNO_3	NH_4Cl Oxidation	N_2	35	7
Tc^{99} Ppt.	HNO_3	Neut. Na_2CO_3	CO_2	1000	70
Oxalate Ppt.	HNO_3	Oxalate Destruction	CO_2	220	7
Ce^{144} Oxidation	$KMnO_4$	Citric Acid destruction	CO_2	111	11
Ce^{144} Reduction	H_2O_2	H_2O_2 Decomposition	O_2	594	60
Magnetite Ppt.	NaOH	Formation of NH_4OH	NH_3	9000	100
Cs^{137} Concentration	None	Radiolytic Hydrogen Generation	H_2	-	0.01
Ce^{144}					
Concentrate Storage	None	Radiolytic Hydrogen Generation	H_2	-	0.06
Evaporation	None	2:1 Concentration	$Ru^{106}O_4$	0	-
STT Shipment	None	Radiolytic Hydrogen Generation	H_2	6	-

Pressure Hazards. The operational procedure requires six inches of water vacuum for all processing which is more than adequate to control the gaseous release from the above reactions.

If an operational error is committed and the chemical addition is increased before or during the above reactions, a pressurized vessel could result. The off-gas system would continue to take out the bulk of the gas liberated. The remainder would vent to the cell ventilation through the pressure relief flapper valve on the solution addition line. If extremely high pressure were encountered, radioactive solution would back up instrument lines, jets and samplers. This equipment is located in cell ventilated and shielded areas and would create a building hazard, but would be contained in the building.

$Ru^{106}O_4$ Gas Hazards. The $Ru^{106}O_4$ gas liberation in the evaporator (E-16) is controlled by the batch evaporation process which controls the acid normality from 3 N to 5 N. Under these conditions no $Ru^{106}O_4$ gas is liberated.

If an operational error were made which drained the maximum volume of 60% HNO_3 from the HNO_3 storage tank into the evaporator, the final concentration would be 7 N and $Ru^{106}O_4$ gas would not be liberated. This is not a credible accident since chemicals are not added to the evaporator under routine conditions and the solution addition system would overflow to the cell floor if the HNO_3 addition line were placed in the evaporator solution addition line by mistake.

The most credible accident concerning $Ru^{106}O_4$ gas liberation would be the accidental addition of potassium permanganate solution to a centrifuge bowl during the dissolving period. The centrifuge bowl would contain a 4 N to 5 N nitric acid solution at 85°C and an activity level as high as 30,000 curies of Ru^{106}. The Ru^{106} activity would be released into the off-gas system as $Ru^{106}O_4$ and the F3P off-gas scrubbers and filters would remove greater than 99% of the activity. A significant but unknown quantity of Ru^{106} would "plate out" on the stainless steel off-gas duct system. The remaining activity would terminate at the 3039 stack area for further decontamination.

The control of this type of accident is effected by careful training of operators in handling and storage of $KMnO_4$. A small stainless steel make-up tank (M-4) in Area 32 has been designated in the Make-Up Procedure(1) as the only tank to be used for $KMnO_4$ make up. This tank is piped directly to N-29 extractor which is the only tank that uses $KMnO_4$ in normal operations. This eliminates the possibility of accidental drainage of $KMnO_4$ into the wrong tank or centrifuge bowl. The responsibility for training the operating personnel as to the hazards of $KMnO_4$ in the F3P system remains with the building supervision.

Hydrogen Gas Generation Hazards. An estimated off-gas flow through each vessel in the building is 280 liters/minute of air. This figure varies depending on the type of vessel in question, but under normal conditions gives more than adequate air sweep for removal of hydrogen by radiolytic generation.

The normal shipment of Redox waste from ICPP takes approximately ten days from the time it is loaded until the time it is unloaded at ORNL. During this period, small quantities of hydrogen gas are being evolved from radiolytic hydrogen generation. The container is sealed with valve-type Snap-tites which prevent the release of this gas and pressures up to 18 psi have been measured on arrival at ORNL.

Explosions

The explosion possibilities at F3P would arise from the following sources:

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(1) Appendix No. 5-q

Solvent-nitric acid reaction
Hydrazine-nitric acid reaction
NH_4NO_3 decomposition
Solvent explosion

Solvent-Nitric Acid Reaction. The solvent possibilities arise from the small quantities of TBP which remain in the F3P feed stream from the metal recovery system, the Amsco-D2EHPA solvents used in the Cell 9 and 10 solvent extraction processes, and the strong nitric acid-TBP mixtures used in the Cell 10 continuous counter-current mixer-settler extraction processes. Feed solutions are normally concentrated by a factor of two to one and in no case is the nitric acid concentration greater than 5 N. The normal steam pressure to the evaporator coil is 60 psi and the maximum evaporation rate is never greater than 40 gallons per hour. An evaporation operating error would have to go undetected for a period of four hours or more before the nitric concentration would reach the stage of possible solvent nitration.

The Amsco-D2EHPA solvents (~40 gallons per batch or 80 gallons per week) used in the rare earth extraction are contacted, separated, back extracted, separated and discharged without heating or concentrating the solvent. Small quantities of solvent could remain in the aqueous phase and could be nitrated under the present process of dissolving Pm^{147} oxalate precipitate on a filter. The quantity of solvent remaining on the filter would be very small and the explosive potential very weak.

The potential hazards in the Cell 10 Pm^{147} separation process result from the use of strong (20 M HNO_3) in the separation of the rare earths. This acid, which is used to induce reflux in the cascade, is pumped directly to a tank in the make-up area from which it is metered to the cascade. Prior to a Ca-Sr run all tanks are emptied and washed so that strong nitric is eliminated from the process equipment. The 20 M HNO_3 tank in the make-up area does not have provision for solution addition (other than distilled water piped directly to the tank) so that the addition of an oxidizable material to the strong acid tank is eliminated. The in-cell process hazards are considered as follows:

1. The concentration of HNO_3 in the aqueous phase in contact with TBP is less than 14 M. Such mixtures are not hazardous but in case of fire could become hazardous. However, an automatic spray system which is actuated by rate-of-temperature rise is installed in the cell.

2. In the separation of rare earths with a TBP-HNO_3 system the promethium is recovered from the strip aqueous in the first cycle and from the raffinate in the second cycle by evaporation of the promethium solution in a continuous evaporator. Since both the strip aqueous and raffinate have previously been contacted with TBP, the possibility exists that TBP could be nitrated in the evaporator to produce an explosive compound. Such an occurrence is not considered credible for reasons that will be mentioned in the following paragraphs.

   To reduce the possibility of TBP reaching the evaporator, the raffinate and strip aqueous stream are washed to remove occluded TBP from the streams. Failure of these washes would allow TBP in

small amounts to reach the evaporator. Failure of instrument control on the cascade weir would partially dump the cascade which might flood the washer and result in some TBP reaching the evaporators.

The degradation of the organic phase by acid hydrolysis in the cascade will result in very small quantities of butyl alcohol being transferred to the evaporator by its solubility in the aqueous stream.

The conditions under which nitric acid will react explosively with TBP on evaporation have been studied and reported.* A specific requirement for such a reaction is reported to be a temperature of at least 150°C.

When TBP reacts with nitric acid at high temperatures and high nitric acid concentrates, the primary reaction product is thought to be butyl nitrate.** At temperatures less than that required to produce butyl nitrate the reaction product of TBP and nitric acid is butyl alcohol.

From the above it seems that the TBP or its degradation product would be found in the evaporator as either butyl alcohol or butyl nitrate depending on the acid concentration and temperature.

The raffinate stream from the second rare earth cycle (10.2 M HNO_3) if continuously evaporated will yield a constant boiling (~122°C) solution of ~15 M HNO_3 (~68% HNO_3). The aqueous strip from the first rare earth cycle (~6 M HNO_3) if continuously evaporated will be ~12.2 M in nitric acid. The total concentration of rare earth nitrates in the evaporator pot will not exceed 2 moles. The maximum boiling point of the nitric acid-rare earth solution of the above concentration will not exceed ~126°C.

Since butyl alcohol boils at 117°C and butyl nitrate at 136° these components should not accumulate because they will be fractionated by steam distillation from the evaporator rapidly at 125°C. Any TBP which reaches the evaporator will be converted to either butyl alcohol or butyl nitrate and evaporate.

The possibility exists that an excessive accumulation of phosphoric acid in the evaporator might raise the boiling point of the solution. However, the temperature control will limit the temperature to 130°C. The maximum temperature obtainable from the steam used to heat the evaporator is 135°C which is not sufficient to decompose the TBP degradation products.***

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*TNX Evaporator Incident, Jan. 12, 1953, DP-25.

**Bruce, F. R., Fletcher, J. M., and Hyman, H. H., eds Process Chemistry. Prog. in Nuclear Energy Series III, Vol. 2, Pergamon Press, New York, 1958, p. 314.

***Op. cit., DP-25.

3. In the Sr-Ca separation the raffinate which is evaporated is ~0.05 M in nitric acid. Therefore, the equilibrium concentration of nitric acid in the evaporator should not exceed 0.5 M. The calculated maximum energy release from such a solution is approximately equal to the energy required to evaporate 0.1% of the solution, however, such a reaction is not considered to be credible.

Hydrazine-Nitric Mixing. Twelve liter quantities per batch of 42.5% hydrazine and 60% nitric acid are used in the F3P mainstream processing. These chemicals are added to the same vessels but during normal operations are compatible due to the high dilution factors.

A maximum credible accident could be postulated (scientific evidence lacking) from the accidental addition of 12 liters of 42.5% hydrazine to a dissolving of a rare earth oxalate precipitate in a centrifuge bowl. The bowl at this time could contain 100,000 curies of Ce^{144} nitrate at 85°C in a volume of 9 gallons of 12 N HNO_3. Pending evidence to the contrary, it is assumed that the explosion could rupture the centrifuge stainless steel casing and overcome the cell ventilation system. The force of the explosion could crack the G-K seal on the cell blocks which would allow contamination to escape into the crane bay area. Other sources of leakage are at the air inlet to each cell, instrument lines, jet lines and possibly manipulator arms in the process control cells 18 and 19.

Under these conditions, the building containment system would remove the contaminated gases through the filter system and then to the 3039 stack area filter systems. The F3P filters would not be expected to suffer damage from this explosion. The building operating area would be contaminated to a high level and could conceivably cause fatalities from overexposure. The building would be untenatable and would be a source of radiation in the building vicinity.

The possibility of this type of accident is not considered credible under present operating conditions and controls. The handling of hydrazine has been the subject of a number of safety meetings and all personnel are aware of its potential hazards. The building foremen are charged with the responsibility of supervising the addition of hydrazine to a process vessel. The dispensing

valve on the 55-gallon drum of 42.5% hydrazine is locked and the foremen have the only keys for operating this valve. They are also responsible for keeping the container locked at all times.

Ammonium Nitrate Decomposition. Ammonium nitrate is formed in the F3P off-gas system by the reaction of ammonia gas and nitric acid fumes from standard operating procedures. The normal process procedures used in the F3P vessel create a considerable amount of condensation in the off-gas lines which keeps the NH_4NO_3 washed out of the system, or at least keeps it from building up to the point where the off-gas lines are plugged with NH_4NO_3.

The most credible accident concerning NH_4NO_3 would occur during a period of maintenance or construction on the off-gas system. A pipefitter cutting into the line, or welding a new nipple to the present off-gas line, could detonate the dried NH_4NO_3 in the system. Under building maintenance procedures, the off gas would be manually valved off at the inlet to the building and the main off-gas line would be unharmed.

The new scrubber systems on the F3P and metal recovery buildings will eliminate the formation of NH_4NO_3 in the main header to the 3039 stack. The formation of NH_4NO_3 in the F3P off-gas header is not controlled at the present time. A periodic clean-up of the off-gas line will be accomplished by boiling water in the process vessels and allowing the steam condensate to flush the system. This is a standard procedure during decontamination periods for any maintenance or construction work in the cells. Welding on off-gas lines has been forbidden unless the line has been thoroughly flushed with hot water.

Solvent Explosion. The possibility of an explosion from solvent vapors during normal operations is not credible. The solvents are not heated or steam jetted and are removed from the system immediately after they have served their purpose. In the case of accidental heating, the off gas would remove the vapors from the tank and effectively condense the gas from the normal off-gas stream.

The possibility of a solvent fire in the process cells is a credible accident. This could result from an electrical short circuit in the cells or a disregard of electrical grounding rules in the solvent handling area. An automatic sprinkling system would be actuated from the heat and would contain the fire.

The contaminated smoke and gases would contain rare earth activities and the cell ventilation system would remove and filter the majority of these fumes. If an explosion occurred from the burning solvent, it would be a low grade type and the force would be contained in the cell.

Radioactive Solution Burping

The maximum accident of this nature would occur in the tank farm fraction tanks containing concentrated rare earth solution with up to 100,000 curies of Ce^{144} plus other rare earth activity.

Under normal conditions, the solution is 2 N to 4 N in nitric acid and kept cooled by the flooded cell, fin cooler technique. If an operator

accidentally jetted a high caustic solution into this tank, the rare earth hydroxides would precipitate and settle to the bottom of the tank. The cooling system would continue to operate but the possibility of a pressure bubble forming in the rare earth precipitate cannot be discounted. This bubble would cause the tank to burp and overcome the normal off gas on the vessel. Radioactive solution would back-up the instrument, sampler, and jet lines which are located in an uncontained and unventilated area on the F3P tank farm.

The burping in a fraction tank is a credible accident, however, the normal safeguards of high temperature and high level alarm systems on these tanks would give adequate warning if the supervisor answering the alarm correctly determines the problem. Also, the fact that the solution going to the wrong tank would be reported to supervision and corrective action could be taken immediately would prevent this type of accident.

Pilot Plant Operations

The F3P is a pilot plant and must adjust its procedures to meet the varying types of feed that are sent to the plant for processing. In the first year of operations, five different types of feed materials were processed and procedures were adjusted while operating on the process solution. A committee has been set up in the Isotopes Division to review all chemical process changes in the production of radioisotopes.

Criticality Hazard

The possibility of a critical solution in the F3P system is a credible assumption. The normal plant data indicate a build-up of uranium and plutonium in the process of concentrating the rare earth fractions. The normal process steps increase the concentration of rare earths per unit volume from the feed material from Building 3019 by a factor of approximately 16. The control of uranium and plutonium entering the F3P depends on the analyses of the 3019 waste stream. These analyses could vary by a factor of ten or higher and could result in a high concentration of plutonium and uranium in the rare earth precipitate in a volume of approximately 20 liters in the centrifuge bowl. The major factor that has to be taken into consideration is the fuel enrichment of the elements processed in the metal recovery system. A combination of plutonium and enriched uranium would be in the centrifuge bowl.

All waste streams into the F3P are analyzed on a composite basis for plutonium and uranium and the building supervisor is responsible for determining the maximum concentration in the rare earth fractions. The uranium and plutonium concentration in the F3P operations are also reviewed by the Criticality Hazards Committee.

Decontamination Hazards

The decontamination solutions[2] at the F3P are added in a specific pattern. The flow of these solutions is controlled by the building supervisor to meet the requirements of the system to be decontaminated.

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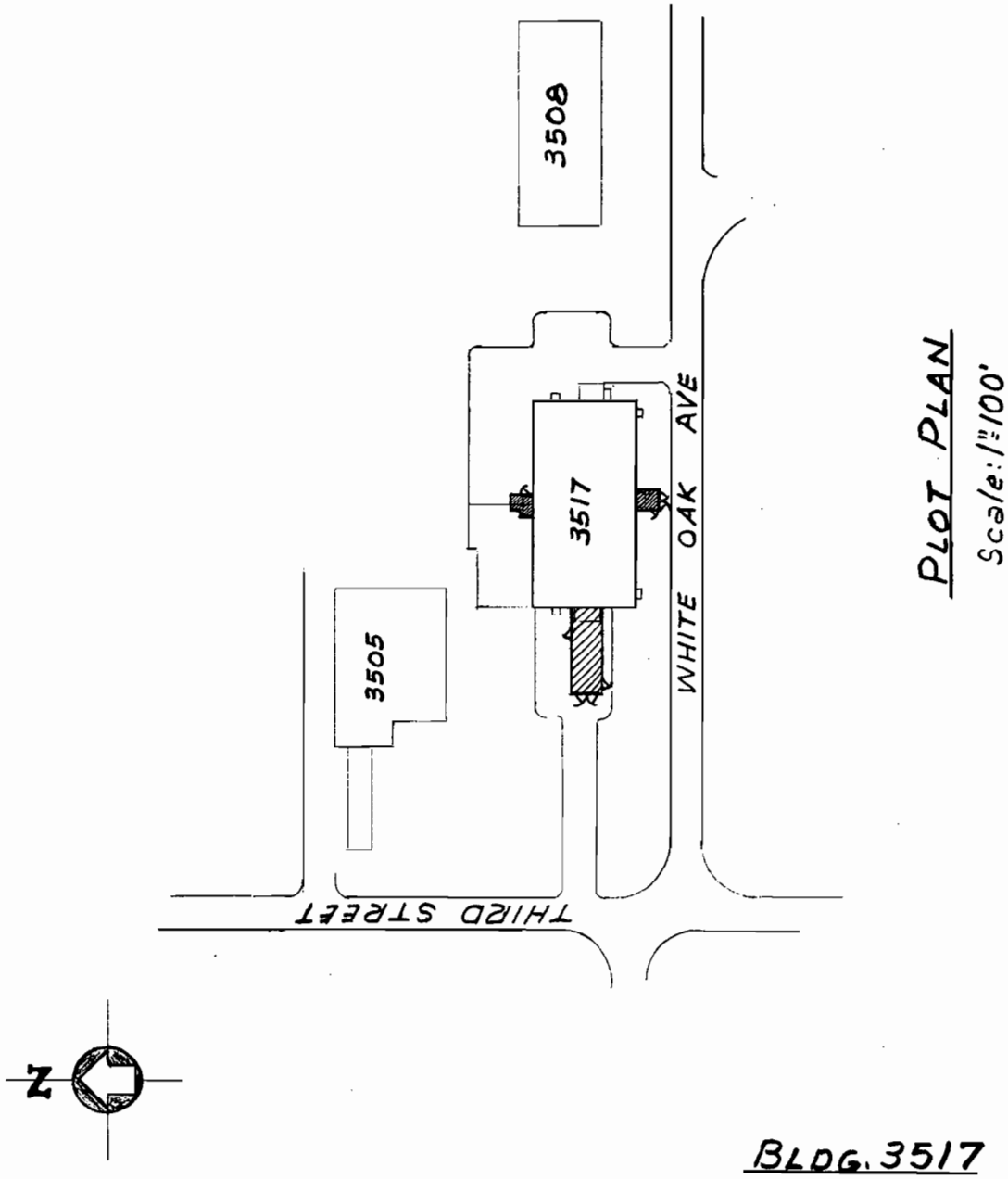
(2) Appendix No. 10

All lines to critical areas or vessels are capped at the panelboard switches to prevent accidental jetting to the wrong vessel.

The major source of a hazardous incident during decontamination is the continuing of routine processing in other cells and the storage of concentrated material in adjacent cells or tank farm area. It is impossible to remove all the concentrated radioisotopes from the building for decontamination procedures.

The greatest safeguard against accidents during decontamination lies in the intelligence and training of the operating personnel and supervision.

APPENDIX NO. 1



PLOT PLAN

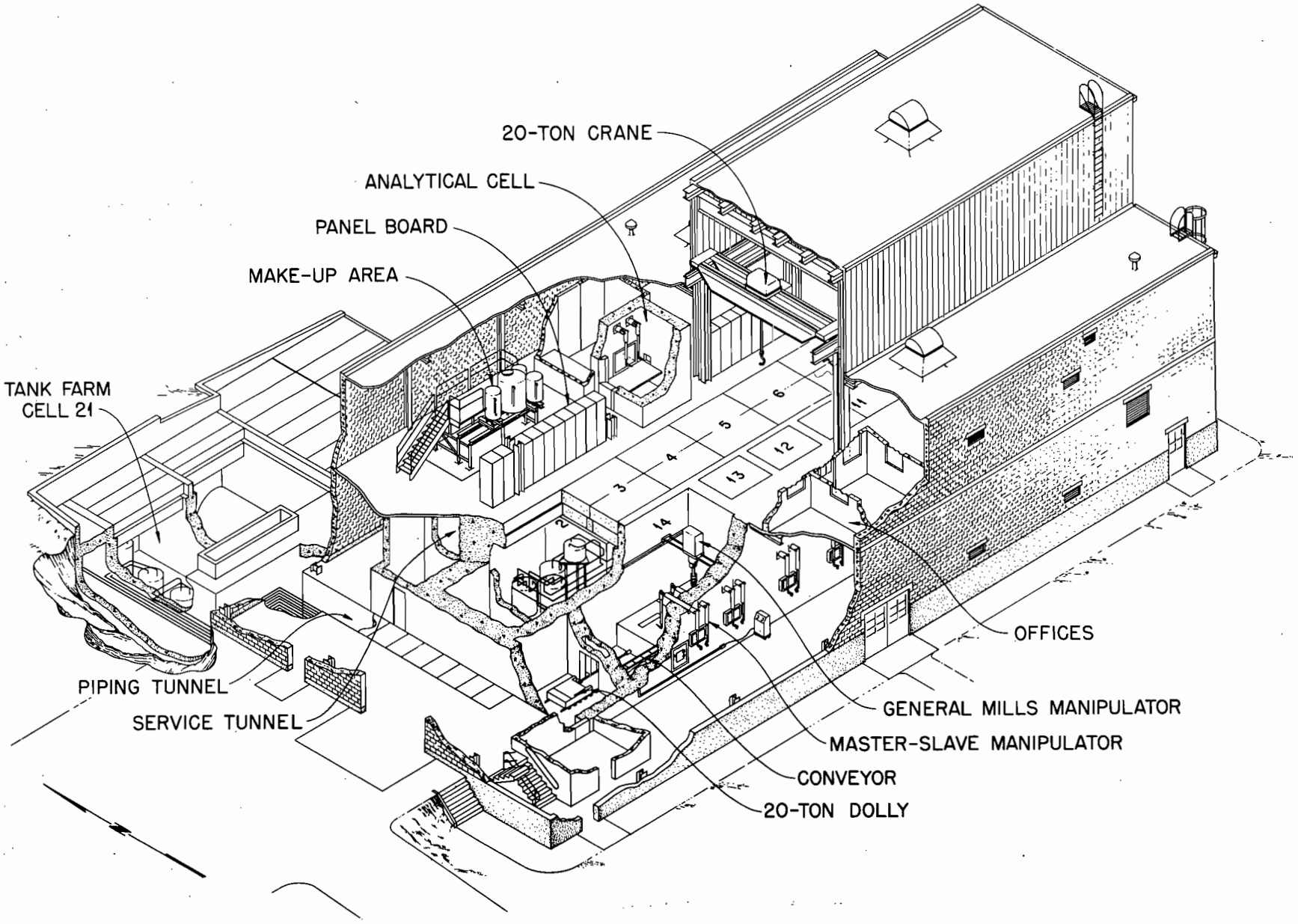
Scale: 1" = 100'

NOT CLASSIFIED

A-38710

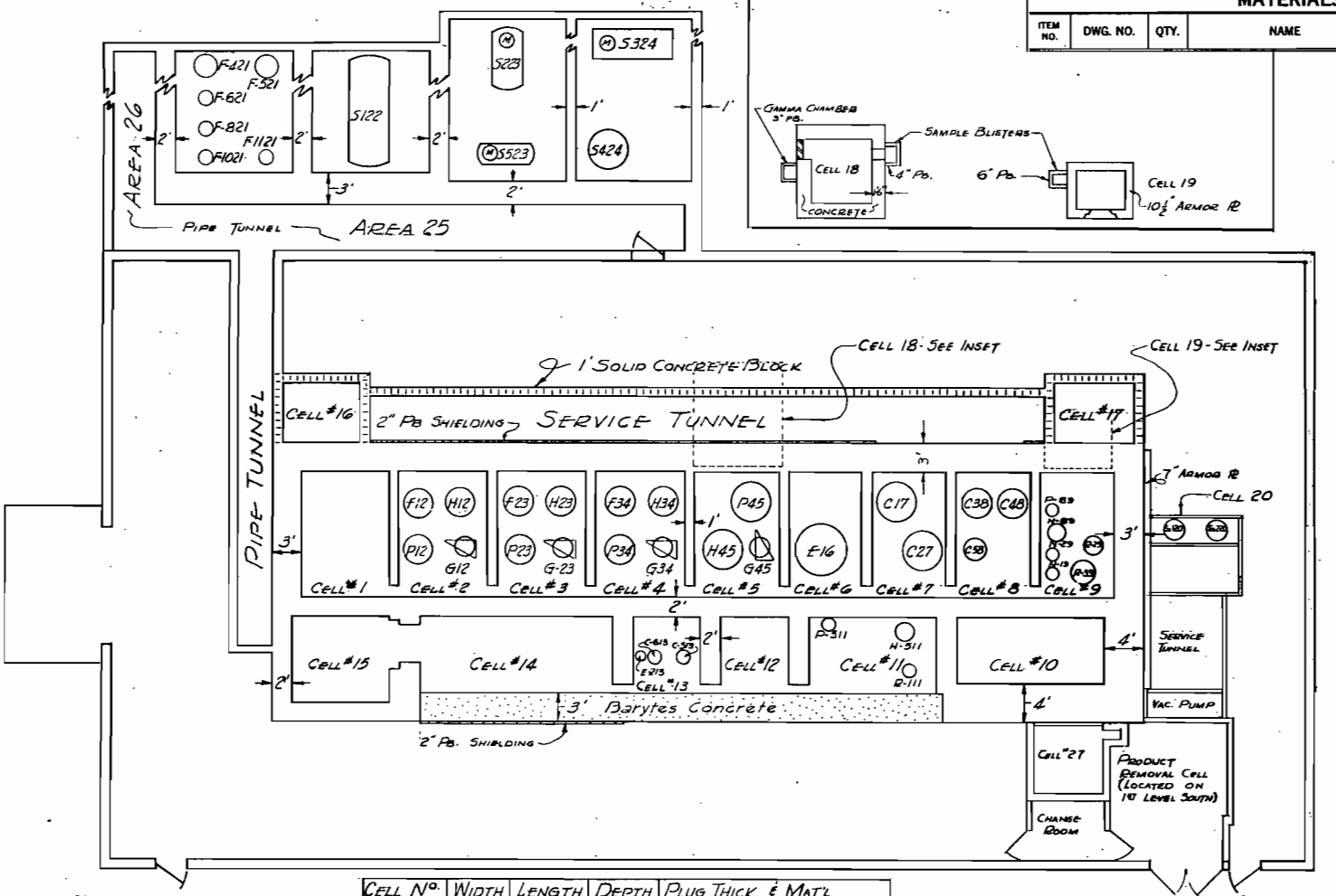
BLDG. 3517
PLOT PLAN
CONTAINMENT FOR
EXPLOSIVE RELEASE IN CELLS
DRAWING A-

Appendix N° 2



MULTICURIE FISSION PRODUCTS PILOT PLANT — OAK RIDGE NATIONAL LABORATORY

APPENDIX NO. 3



1. Cell Liners - 3/16" 347 Stainless Stl.
2. Cell Doors - Solid mild stl.
3. Sliding Door - Bottom - steel, Top - concrete filled
4. Cell 19 Shielding - 10½" Armor PL
5. Cell 20 Shielding -
6. Cell 27 Shielding - 9" Armor PL

CELL No.	WIDTH	LENGTH	DEPTH	PLUG THICK. & MAT'L
1,2,3,4,5	9'-0"	12'-6"	12'-0"	4'-0" CONCRETE + 3½ A. PL
6,7,8,9	7'-6"	12'-6"	12'-0"	4'-0" " " "
10	15'-0"	7'-0"	14'-0"	4'-0" "
11	13'-0"	8'-0"	14'-0"	4'-0" " + 3½" A. PL
12, 13	7'-0"	8'-0"	14'-0"	4'-0" " " "
14	20'-0"	8'-0"	15'-0"	3'-0" (Barytes) " "
15	10'-0"	9'-0"	16'-0"	2'-0" CONCRETE
16, 17	8'-0"	6'-0"	14'-0"	1'-0" "
21, 22	12'-0"	25'-0"	10'-0"	4'-0" " + 3½" A. PL
23, 24	12'-0"	30'-0"	12'-0"	2'-0" " " "
18	6'-0"	6'-6"	10'-6"	2'-0" " -
19	5'-0"	4'-0"	10'-6"	0'-3½" A. PL
20	6'-6"	7'-6"	12'-0"	3½" A. PL -
27	7'-0"	7'-0"	10'-6"	10½" A. PL

MATERIALS LIST

ITEM NO.	DWG. NO.	QTY.	NAME	SIZE	MATERIAL

NO.	REVISIONS	DATE	APPR.	APPR.
A	CELLS 18, 19, 20, & 27 ADDED			
	PB ADDED TO SERVICE TUNNEL	1-22-60	BC	

DRAWING NO. C-RD-1176

DRAWN	DATE	CHECKED	DATE	APPROVED	DATE
EARLY	19/11/57				
DESIGNED	DATE	SUBMITTED	DATE	APPROVED	DATE

HEALTH PHYSICS	MEDICAL	OPERATIONS	SAFETY
FIRE PROTECTION	RESEARCH SHOP	MAINTENANCE	

THIS DRAWING CLASSIFIED AS UNCLASSIFIED PER Eugene Lamb

SCALE: 1/8" = 1'-0"

REFERENCE DRAWINGS	DWG. NO.

BLDG. 3517

CELL DIMENSIONS

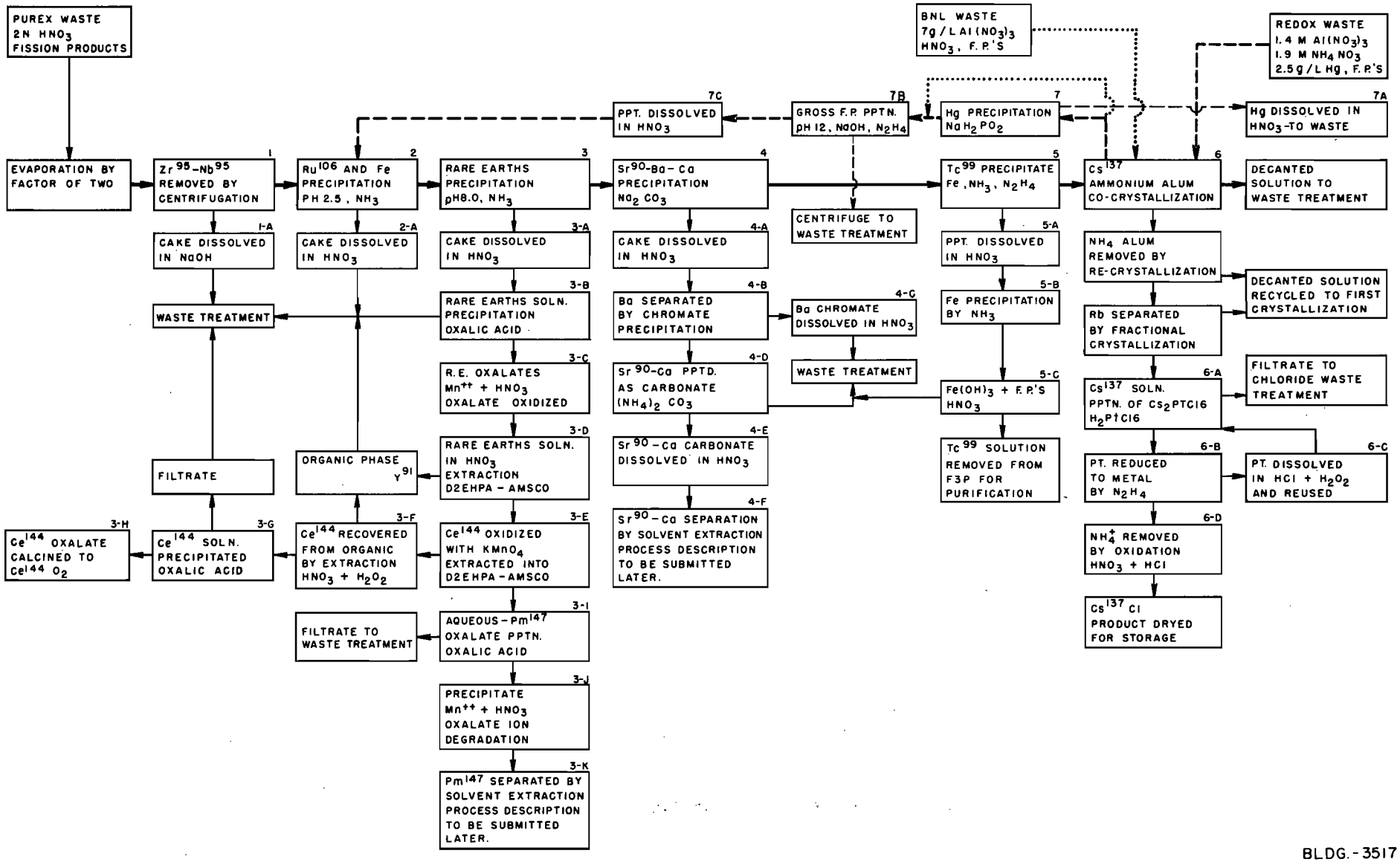
OAK RIDGE NATIONAL LABORATORY
OPERATED BY
UNION CARBIDE NUCLEAR COMPANY
A DIVISION OF UNION CARBIDE AND CARBON CORPORATION
OAK RIDGE, TENNESSEE

SUBMITTED	ACCEPTED	APPROVED

C-RD-1176 REV. A

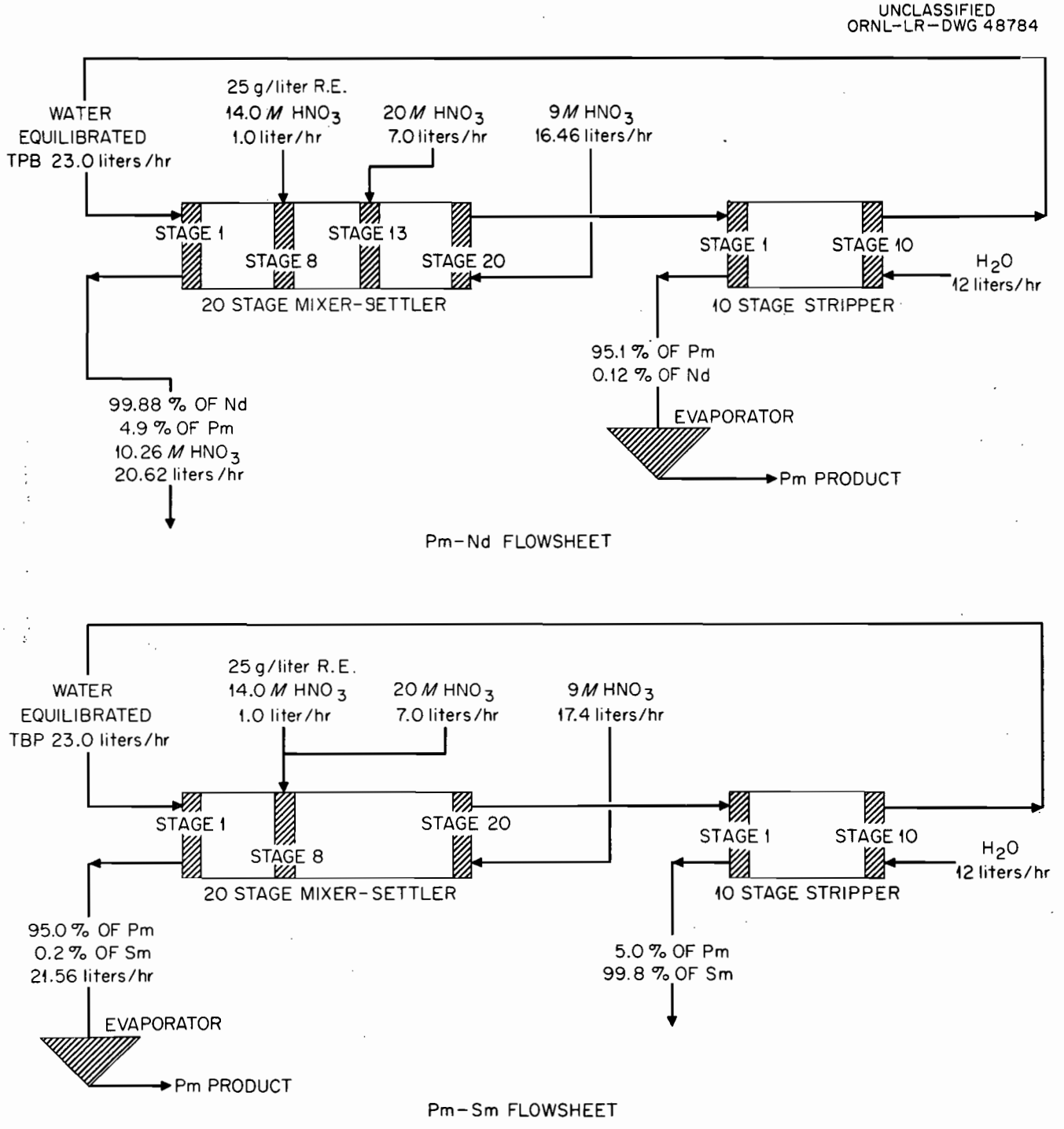
APPENDIX NO. 4

TYPICAL CHEMICAL PROCESSES IN F 3 P



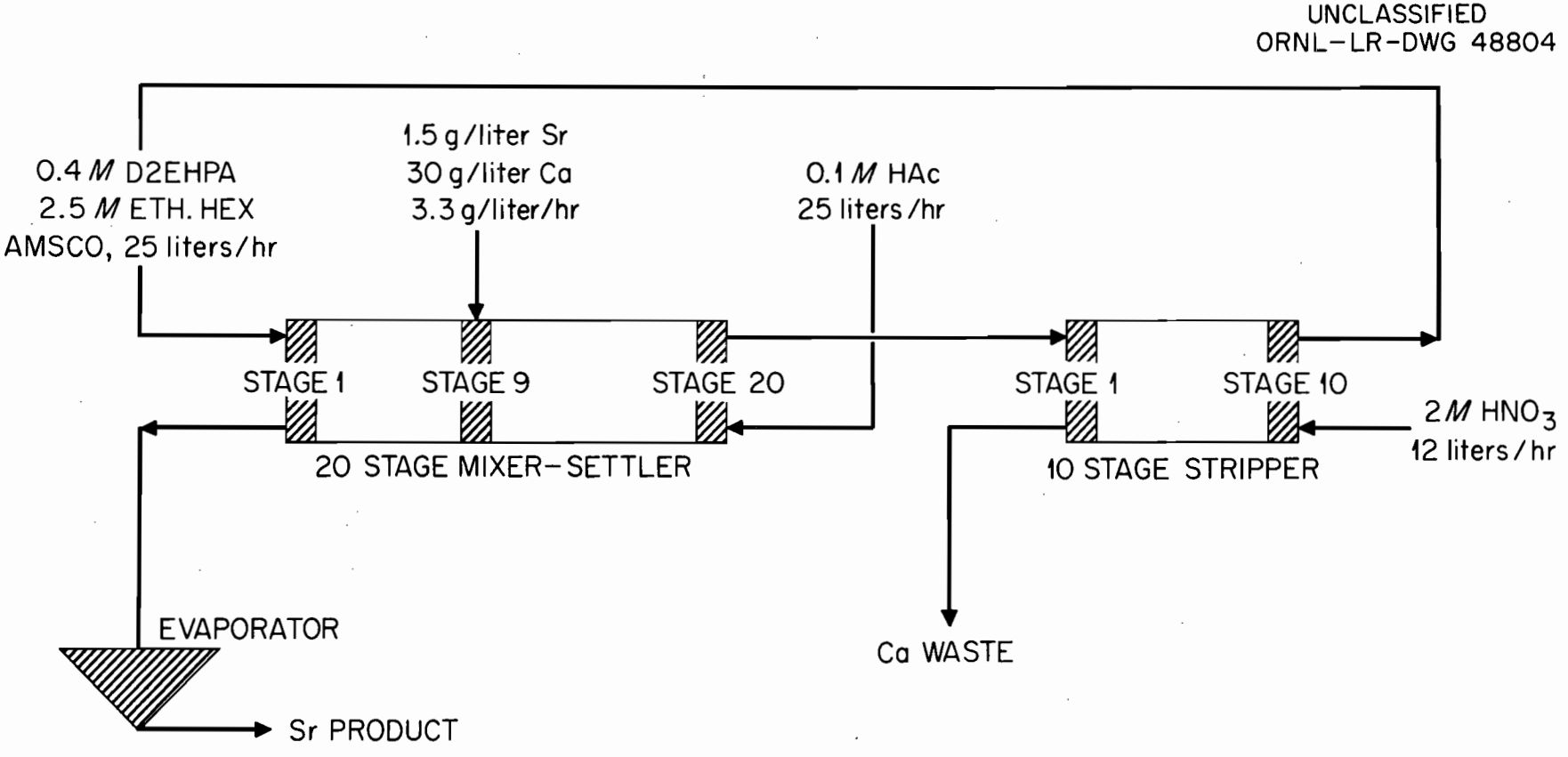
BLDG.-3517
B-38570

APPENDIX 4-A



Overall Flowsheet for the Separation and Purification of Pm^{147}

APPENDIX 4-B



Ca-Sr Flowsheet.

APPENDIX NO. 5-a

CRYSTALLIZATION PROCEDURE

I. Redox and 3019 Feed Crystallization

A. Chemical Make-Up

1. _____lbs. sodium sulfate (Na_2SO_4)

2. _____lbs. ammonium sulfate (NH_4SO_4)

3. _____gals. 60% nitric acid (HNO_3)

B. Operational Checks

1. All process, service, solid addition and solution addition valves are closed.

2. All valves to and from Pump U-125 are closed at P.B. No. 3.

3. All valves to and from W-19 and W-20 jet are closed at P.B. No. 3.

4. C-17 tank is empty.

5. C-17 has at least 6 to 8 inches of off gas.

6. Close all valves in W-19 and W-20 valve pit.

C. Operations for W-19 and W-20 Solution

1. Jetting Procedure for W-19 and W-20 Tanks

(a) Open the manual valve at W-19 and W-20 valve pit to the tank from which solution will be pumped.

(b) Open the two valves on the discharge side of W-19 and W-20 to C-17 jet.

(c) Open the valve on the suction side of W-19 to W-20 to C-17 jet.

(d) Turn on the steam to W-19 and W-20 to C-17 jet.

(e) Turn on C-17 contact microphone, agitator and process water to coils.

(f) Jet_____gals. of solution to C-17 tank and then close all valves to and from W-19 and W-20 jets.

(g) Follow Crystallization Operation Procedure.

II. Purex Crystallization

A. Chemical Make-Up

1. 0.5 to 1.0 lb./gallon ammonium alum

2. 20 gals. 70% nitric acid

3. _____ lbs. water

B. Operations

1. Add 20 gallons 60% nitric acid to C-17 crystal bed.

2. Jet solution from H-45 to C-17 while controlling the off gas and holding temperature below 40°C.

III. Crystallization Operations

1. Add required chemicals to crystallizer calculated to give 375 gallons in C-17 and C-27 or 100 gallons in C-38 and C-48.

2. Turn on crystallizer agitator and contact microphone.

3. Heat solution to 90°C.

4. Digest solution at 90°C for 15 minutes.

5. Record crystallizer volume data with agitator off.

6. Turn on crystallizer agitator.

7. Heat crystallizer sampler jacket, then sample hot solution. Code - Batch No. C Tank No. Csl Record gamma chamber reading.

8. Turn on process water to crystallizer coils.

9. Turn on air to crystallizer filter blowdown.

10. Cool to 40°C with the process water. Turn off process water.

11. Turn on chilled water to crystallizer and cool until temperature break. Record temperature break.

12. Continue cooling crystallizer to 5°C and hold at that temperature.

13. Continue agitating crystallizer solution for 30 minutes from temperature break time.

14. Determine crystal volume from chart, sample, or lab crystallizer.

15. Check liquid level on the tank designated to receive filtrate. If empty, proceed with next step.

16. Turn off crystallizer agitator and filter blowdown air.

17. Settle the crystal bed for 15 minutes.

18. Filter crystallizer solution to designated tank.

19. Turn off chilled water, agitator and contact microphone to crystallizer.

20. Crystallizer is ready for next batch of feed solution or filtrate from primary crystallizer.

NOTE: Control all crystal beds to 5% of the operating volume of the tank. If the gamma chamber indicates more than 500 curies of Cs^{137} activity in C-27 or C-48, dissolve the bed and return to C-17 at the first opportunity.

Crystal bed transfers to C-38 and C-5 should be reduced in crystal volume to 2% at the tank operating volume.

IV. Crystal Bed Removal

A. Operation

1. Add _____ gallons of water to crystallizer.

2. Turn on agitator and contact microphone.

3. Heat solution to 90°C.

4. Digest solution at 90°C for 20 minutes.

5. Turn on the process water to crystallizer coil and turn on the air to filter blowdown.

6. Cool to 60°C and hold at that temperature.

NOTE: Watch for a crystal temperature break. If crystals form, a recrystallization must be made.

7. Check that designated receiver crystallizer is empty except for crystal bed.

8. Turn on transfer jet.

9. When crystallizer is empty, turn off the steam to transfer and cut off the air to blowdown filter.

10. Turn off agitator.

11. If receiving crystallizer volume is less than operating volume, add water as specified by supervision to bed crystallizer and repeat Steps (1) through (5).

APPENDIX No. 5-b

MERCURY PRECIPITATION PROCEDURE (Hgl)

Mercury Precipitation

1. Chemical Make-Up for P-12 and G-12

   (a) 2.0 gallons of 60 percent HNO_3 (nitric acid) in M-331

   NOTE: On each third batch, an additional 6.0 gallons of 60 percent HNO_3 is required.

   (b) 6.0 gallons of 4.0 M Na H_2PO_2 (sodium hypophosphite) make up = 20.0 pounds of Na H_2PO_2 dissolved in 6.0 gallons of water.

2. Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Turn on contact microphone, agitator and process water to jacket.

   (d) Jet from S-122 to precipitator until a maximum of 150 gallons is reached.

   (e) Record precipitator volume with the agitator off.

   (f) Turn on the agitator and if requested, sample solution. Code = ___Hgl.

   (g) Adjust the solution to 0.3 N. Free acid by adding 19 M caustic or 60 percent HNO_3 (nitric acid). The amount of 70 percent HNO_3 necessary will be determined by laboratory analysis for free acid.

   (h) Follow with a 2.0 liter water rinse.

   (i) Check that solution temperature is 30°C or less.

   (j) Slowly add 6.0 gallons of 4.0 M Na H_2PO_2 (sodium hypophosphite) to precipitator which should make the solution 0.1 M in sodium hypophosphite. Follow with a 2.0 liter water rinse.

   (k) Turn off jacket water.

   (l) Heat solution to 60°C by setting the temperature controller at 60°C and turn on reflux condenser water.

   (m) Digest solution at 60°C for 1.0 hour.

   (n) Turn off the inlet steam valve to jacket by setting the steam controller at 0°C. Close the outlet steam valve.

   (o) Turn on the process water to precipitator jacket and cool solution to 30°C. Leave jacket water on until precipitator is empty.

(p) Digest solution for one hour at 30°C or lower

(q) Sample precipitator solution for a mercury test.

Draw up 10 ml of slurry. Centrifuge 5 minutes. Pour off 5 ml of supernate to a clean test tube or sample bottle. Add 1 ml of 2.0 N HNO_3. Add 2 ml of 1.0 M Na H_2PO_2 (sodium hypophosphite) to the solution. Mix the solution briefly. Wait 2 minutes. If no finely divided black mercury metal forms, the test for mercury is negative and the slurry may be centrifuged. If the test for mercury is positive, add 2.0 gallons of 4.0 M NaH_2PO_4 and repeat Steps (k) through (q).

(r) If requested, take A and B samples at this point.

(s) Record percent and volume of precipitate.

3. Mercury Precipitation Centrifugation

(a) Check that hold-up tank is empty and all service valves are closed.

(b) Check that all valves servicing the centrifuge are closed.

(c) Place skimmer arm in the "OUT" position.

(d) Turn on the D.C. brake switch.

(e) Turn on oil pump to centrifuge bearings. Check centrifuge oil supply tank level.

(f) Turn on contact microphone.

(g) Turn on centrifuge motor.

(h) When tachometer reaches 1750 RPM, adjust the control jet to allow a 1.5 G.P.M. flow from precipitator to centrifuge and check the following every 30 minutes:

1. Oil pump operating.
2. Oil supply level.
3. Centrifuge motor temperature.
4. Control jet flow rate.
5. Precipitator temperature is 20°C to 30°C.

(i) When precipitator is empty, turn off the control jet to centrifuge.

(j) Turn off agitator, jacket water and reflux condenser water.

(k) Turn off centrifuge motor and proceed with magnetite precipitation procedure.

NOTE: If third batch, leave centrifuge running.

(l) Record the number of batches which have collected in centrifuge bowl.

(m) Hold the precipitate in the bowl until three batches have collected.

4. Mercury Precipitate Dissolving

DO THE FOLLOWING STEPS A THROUGH V EACH TIME THAT THREE BATCHES OF PRECIPITATE HAVE COLLECTED IN CENTRIFUGE

(a) With centrifuge running and the D.C. brake switch on, slowly skim to Notch 7. Skimming should be done over a period of 5 minutes.

(b) When skimming is finished, place skimmer arm in the "out" position.

(c) Add 5.0 gallons of water to precipitator.

(d) Jet the solution to the centrifuge, then turn off the jet.

(e) Slowly skim the centrifuge to Notch 7. Skimming should be done over a period of 5 minutes.

(f) When skimming is finished, place skimmer arm in the "out" position.

(g) Repeat Steps (c) through (f).

(h) Turn off centrifuge motor.

(i) Add 6 gallons of 60 percent HNO_3 (nitric acid) to centrifuge. Follow with a 500-milliliter water rinse.

(j) Turn on the steam to coil by setting the steam controller to 100 lbs. per hour of steam and heat to 85°C.

(k) Hold solution temperature between 80°C and 85°C.

(l) Turn on brake switch and job centrifuge to 1750 RPM. Then turn off centrifuge.

(m) Repeat the jogging procedure and check the following every 30 minutes for one hour:

1. Steam rate.
2. Solution temperature.
3. Centrifuge motor temperature.

(n) Turn off the steam to centrifuge coil by setting the steam controller to 0 lbs./hr.

(o) Turn on the process water to coil.

(p) Cool solution to 60°C. Then turn off the water to coil.

(q) Jet solution to designated fraction tank.

(r) Add 5.0 gallons of water to centrifuge.

(s) Jog to 1750 RPM, then shut off.

(t) Jet solution to designated fraction tank.

(u) Repeat Steps (r) through (s).

(v) Jet second water wash to S-324.

APPENDIX No. 5-c

MAGNETITE PRECIPITATION PROCEDURE

Magnetite NaOH Precipitation of Fe, AE, RE, Ru, Sr and Tc

1. Chemical Make-Up

   (a) 30 gallons of 19 M NaOH (caustic) in M-231 Tank.

   (b) 4.0 pounds of Na_2CO_3 (sodium carbonate) dissolved in 3.0 liters of water.

   (c) 3 gallons of 60 percent HNO_3 (nitric acid) in Tank M-331.

   (d) 6 liters 42.5 percent hydrazine hydrate solution.

2. Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) If Hg precipitation procedure used, jet solution from H-12 to precipitator. If 3019 solution, jet _____ gallons to precipitator.

   (d) Turn on the precipitator, contact microphone and agitator when liquid level reaches 20 percent.

   (e) With agitator off, record volume on data card.

   (f) (1) Turn on precipitator agitator and sample solution. Code ____Mgl.

   (2) Add 15 gallons 19 M NaOH while holding temperature at 50°C. Check for precipitate. If there is a precipitate, add one gallon HNO_3 and digest at 90°C until solution is clear.

   (g) Heat precipitator to 70°C. Add 6.0 liters of 42.5 percent hydrazine hydrate and a one-liter water rinse to precipitator. Digest 15 minutes at 70°C. Take 10 ml sample for iron precipitation test. Record digestion time and temperature. Details of the iron test are as follows:

Test for Precipitation of Iron as Magnetite on Main Stream Slurry in Precipitator

Draw up 5 ml of solution from precipitator. The solution should be clear at this point. Add an equal volume of 25% caustic (10.0 N) to this solution. The heavy white voluminous precipitate of aluminum hydroxide that forms should redissolve in the excess caustic. A small amount of black precipitate of magnetite (Fe_3O_4) should remain suspended in the excess caustic liquor. There should be no orange color due to the presence of ferric hydroxide ($Fe(OH)_3$) at this point. Centrifuge the slurry and note the percent of precipitate. The percent of precipitate should be about

2.0% based on the original solution. If an orange color is noted or if the percent precipitate is unusually large, notify supervision. Otherwise, you can proceed with the run.

(h) Add 35 gallons of 19 M NaOH (caustic) to precipitator. Add the rest of the 19 M NaOH (caustic) more rapidly over a period of 15 minutes.

(i) Add 4.0 lbs. of Na_2CO_3 (sodium carbonate) dissolved in 3.0 liters of water to precipitator. Follow with a 1.0 liter water wash.

(j) Turn off precipitator jacket water.

(k) Turn on the process water to reflux condenser.

(l) Open the inlet steam valve to jacket by setting the temperature controller at 90°C and hold at that temperature.

(m) Sample solution for precipitate volume.

(n) Add the necessary amount of 19 M NaOH (caustic) to remove $Al(OH)_3$ from precipitate volume and until two consecutive samples show the same precipitate volume. Record final precipitate volume in gallons and volume of 19 M NaOH added.

(o) After the desired percent precipitate is reached, digest the solution for an additional 30 minutes at 90°C.

(p) Turn off the steam to jacket by setting the temperature controller at 0°C, then close the outlet steam valve.

(q) Turn on the process water to jacket and cool solution to 20°C. Leave the cooling water on jacket until precipitator is empty.

(r) When required, take an A and B sample for the laboratory with A as the supernate and B as the precipitate.

3. Alkaline Earths and Rare Earths Precipitate Centrifugation

(a) Check that hold-up tank is empty and that all service valves are closed.

(b) Check that all valves servicing the centrifuge are closed.

(c) Set centrifuge skimmer in the "out" position.

(d) Turn on the D.C. brake switch.

(e) Turn on the oil pump to centrifuge bearings. Check the centrifuge oil supply tank level.

(f) Turn on centrifuge contact microphone.

(g) Turn on centrifuge motor.

(h) When the tachometer reaches 1750 RPM, turn on the control jet from the precipitator to the centrifuge at a flow rate of 1.0 to 1.3 G.P.M. and check the following every 15 minutes:

1. Oil pump operation.
2. Oil supply level.
3. Centrifuge motor temperature.
4. Control jet rate G.P.M.

(i) When the precipitator is empty, turn off the control jet.

(j) Turn off the agitator when volume reaches 10%, jacket water and reflux condenser water.

(k) Slowly skim to Notch 7 on the centrifuge. Skimming should be done over a period of 5 minutes.

(l) When skimming is finished, place the skimmer arm in "out" position.

(m) Continue running centrifuge until the water wash is completed.

(n) Add 5 gallons water to the centrifuge bowl.

(o) Five minutes after water addition, slowly skim to Notch 7 on centrifuge. Skimming should be done over a period of 5 minutes.

(p) When skimming is finished, place skimmer arm in the "out" position.

(q) Turn off centrifuge.

4. Rare Earths and Alkaline Earths Precipitate Dissolving

(a) Add 6.0 gallons of 7.0 N HNO_3 (nitric acid) to centrifuge. (Make-up: add 3.0 gallons of 60% HNO_3 to centrifuge. Then add 3.0 gallons of water.)

(b) Turn on the steam to centrifuge coil by setting the steam controller to 100 lbs./hr. of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 30 minutes for two hours and check the following:

1. Steam rate.
2. Solution temperature.
3. Centrifuge motor temperature.

(f) Turn off the steam to centrifuge by setting the steam controller to 0 lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil.

(i) Check that designated fraction tank is ready to receive solution.

(j) Jet solution to designated fraction tank.

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

APPENDIX No. 5-d

BATCH EVAPORATION PROCEDURE - PUREX

1. Operation

   (a) Check that all jet and service valves to E-16 are closed.

   (b) Check that E-16 is ready to receive incoming feed.

   (c) Check that off gas is 6 to 8 inches.

   (d) Open valves from designated tank at W-19 and 20 valve pit to E-16 tank.

   (e) Open valves at PB No. 3 from W-19 or 20 through jet to E-16 tank.

   (f) Turn on W-19 or 20 jet to E-16.

   (g) Turn on D-56 automatic condenser water controller at 50°C.

   (h) Jet 300 gallons of feed to E-16.

   (i) Turn on steam to E-16 coil.

   (j) Continue concentrating W-19 or 20 solution until a concentration of 2:1 has been achieved. Record volumes at Panel Board.

   (k) Cool E-16 to 60°C and cut off coil water.

   (l) Check that P-12 is ready to receive a batch.

   (m) Jet 150 gallons into P-12. Use P-12 liquid level for this measurement.

   (n) Repeat batch evaporation procedure.

APPENDIX No. 5-e

ZIRCONIUM-NIOBIUM PRECIPITATION PROCEDURE (CODE Zr-1)

Centrifugation of Insoluble Zr-Nb-Si Precipitate

1. Chemical Make-Up

   (a) 1.0 gallon 19 M NaOH for dissolving.

   (b) 1.0 gallon 60% HNO_3 for dissolving every fifth batch.

2. Precipitation Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Turn on contact microphone and process water to jacket.

   (d) Jet 150 gallons from E-16 to precipitator. Do not exceed a maximum of 150 gallons in precipitator. When liquid level reaches 20%, turn on agitator.

   (e) Record precipitator volume with agitator off.

   (f) If sample is requested, turn on agitator and sample solution. Code ___Zr-1.

   (g) Cool P-12 to 30°C.

3. Centrifugation Operation

   (a) When temperature reaches 30°C, start centrifugation operation.

   (b) Continue cooling P-12 during the centrifugation operation until the temperature reaches 20°C, then cut off jacket water.

   (c) Check that hold-up tank is empty and all service valves are closed.

   (d) Check that all valves servicing the centrifuge are closed.

   (e) Place skimmer arm in "out" position.

   (f) Turn on D.C. brake switch to the centrifuge.

   (g) Turn on oil pump to the bearings. Check centrifuge oil supply tank level.

   (h) Turn on the contact microphone.

   (i) Turn on the centrifuge motor.

   (j) When the tachometer reaches 1750 RPM, turn on control jet from the

precipitator to the centrifuge, adjust the steam pressure regulator to give a flow of 1.0 gallon per minute to the centrifuge.

(k) Check the following every 30 minutes:

1. Oil pump operating.
2. Oil supply level.
3. Centrifuge motor temperature.
4. Control jet flow rate.
5. P-12 temperature is 20°C to 30°C.

(l) When liquid level reaches 10%, turn off agitator.

(m) When the precipitator is empty, add two 5-gallon water washes to precipitator and centrifuge. Turn off the control jet.

(n) Turn off the precipitator jacket water.

(o) Slowly skim to Notch 7 on centrifuge. Skimming should be done over a period of 5 minutes.

(p) When skimming is finished, place skimmer arm in "out" position.

(q) Turn off centrifuge motor.

(r) Record the number of batches collected in centrifuge bowl.

4. Zr-Nb-Si Precipitate Dissolving

(a) Add 5.0 gallons of 4.0 N NaOH to centrifuge. (Make-up: add 1 gallon of 19 M NaOH to centrifuge. Then add 4.0 gallons of water.)

(b) Turn on steam to centrifuge coil by setting the steam controller to 100 lbs./hr. of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 30 minutes for 2 hours and check the following:

1. Steam rate.
2. Solution temperature.
3. Centrifuge motor temperature.

(f) Turn off the steam to centrifuge by setting steam controller to 0 lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil.

(i) Check that F-12 is ready to receive solution and cooling water is on F-12.

(j) Jet solution to designated fraction tank.

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

(n) Repeat Steps (k) through (m).

5. Every Fifth Batch do a Nitric Dissolving in G-12

(a) Add 1.0 gallon 60% HNO_3 and 5.0 gallons of water to G-12.

(b) Turn on steam to centrifuge coil by setting steam controller to 100 lbs./hr. of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 30 minutes for 2 hours and check the following:

1. Steam rate.
2. Solution temperature.
3. Centrifuge motor temperature.

(f) Turn off the steam to centrifuge by setting the steam controller to 0 lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil.

(i) Check that designated fraction tank is ready to receive solution.

(j) Jet solution to designated fraction tank.

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

(n) Jet F-12 to S-122 every fifth batch.

APPENDIX No. 5-f

IRON-RUTHENIUM PRECIPITATION PROCEDURE

Gaseous NH_3 Precipitation of Fe, Ru, Zr and Nb

1. Chemical Make-Up

   (a) 29 gallons 19 M NaOH in M-231 tank.

   (b) NH_3 cylinder on line.

   (c) 3 gallons 60% HNO_3 for dissolving precipitate.

2. Precipitation Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Turn on contact microphone, agitator and process water to jacket.

   (d) Jet 125 gallons from F-1121 to precipitator. Do not exceed a maximum of 125 gallons in precipitator.

   (e) Record precipitator volume with the agitator off.

   (f) Turn on agitator and sample solution. Code ____ Ru2S. Have lab rush acid normality.

   (g) Check that the process water is on to the precipitator jacket. Adjust precipitator solution to 0.2 N acid by the slow addition of 19 M NaOH. Watch off gas! Hold temperature at 50°C.

   NOTE: Sample solution at the end of NaOH addition and check for color. The solution should have no reddish-brown precipitate or color. If solution is colored, add 2 gallons of 60% HNO_3 and digest at 90°C until reddish-brown color has disappeared. Repeat Step (g) and check supervision for volume problems in following operations.

   (h) Record the total volume of 19 M NaOH added to the precipitator.

   (i) Rinse solution addition line with 2 liters of water.

   (j) Heat precipitator to 90°C ±2°C.

   (k) Turn on NH_3 gas flow to precipitator at 1.0 cfm and hold temperature at 90°C ±2°C for 30 minutes.

   (l) Adjust pH probes with buffer solutions.

   (m) Sample solution with cooling water on sampler jacket. Check pH

of solution periodically until it reaches 0.8. Reduce NH_3 flow to 0.5 cfm.

(n) When pH reaches 1.5, cut NH_3 flow to 0.2 cfm and sample solution every 5 minutes until pH reaches 2.5.

(o) When pH is 2.5, immediately turn off NH_3 gas flow. Check final pH and record. pH should be 2.5 to 3.0.

(p) Determine precipitate volume on final sample and record. If precipitate volume is greater than 5.0 gallons, check supervision. If ____ Rul A and ____ Rul B samples are requested, take at this point.

(q) Cool solution to 30°C.

3. Centrifugation Operation

(a) Adjust the steam pressure supplying the control jet to give a flow of 1.0 to 1.3 gallons per minute to the centrifuge.

(b) Check that the hold-up tank is empty and all service valves are closed.

(c) Check that all valves servicing the centrifuge are closed.

(d) Place skimmer arm in "out" position.

(e) Turn on the D.C. brake switch to the centrifuge.

(f) Turn on the oil pump to the bearings. Check the centrifuge oil supply tank level.

(g) Turn on the contact microphone.

(h) Turn on the centrifuge motor.

(i) When the tachometer reaches 1750 RPM, turn on the control jet from the precipitator to the centrifuge.

(j) Check the following every 15 minutes:

1. Oil pump operating.
2. Oil supply level.
3. Centrifuge motor temperature.
4. Control jet flow rate.

(k) When the precipitator is empty, add two 5-gallon water washes to P-34 and centrifuge. Turn off the control jet.

(l) Turn off the agitator and jacket water.

(m) Slowly skim to Notch 7 on the centrifuge. Skimming should be done over a period of 5 minutes.

(n) When skimming is finished, place the skimmer arm in "out" position.

(o) Turn off centrifuge motor.

(p) Record the number of batches collected in centrifuge bowl.

4. Iron-Ruthenium Precipitate Dissolving

(a) Add 6.0 gallons of 8.0 N HNO_3 (nitric acid) to centrifuge. (Make-up: add 3.5 gallons of 60% HNO_3 to centrifuge. Then add 2.5 gallons of water.)

(b) Turn on the steam to centrifuge coil by setting the steam controller to 100 lbs/hr. of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 30 minutes for 4 hours and check the following:

1. Steam rate.
2. Solution temperature.
3. Centrifuge motor temperature.

(f) Turn off the steam to centrifuge by setting the steam controller to 0.lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil

(i) Check that F-34 is ready to receive solution.

(j) Jet solution to designated fraction tank.

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

(n) Repeat Steps (k) through (m)

APPENDIX NO. 5-g

RARE EARTH PRECIPITATION PROCEDURE

Gaseous NH_3 Precipitation of R.E.'s

1. Chemical Make-up

   (a) NH_3 cylinder on line.

   (b) 6.0 gallons 5 N HNO_3 for dissolving precipitate.

2. Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Jet Ru-2 centrifugate from hold-up tank to RE-2 precipitator.

   (d) Turn on precipitator, contact microphone and agitator.

   (e) With agitator off, record volume on data card.

   (f) Turn on precipitator agitator and sample solution. Code RE-2.

   (g) Turn on steam to jacket and heat to 90°C.

   (h) Turn on NH_3 gas flow to < 0.2 cfm and hold at 90°C ±2°C.

   (i) Adjust pH probes with buffer solutions.

   (j) Sample solution with cooling water on sampler jacket. Check the solution periodically for a white precipitate.

   (k) After pH of 7.0 is obtained, raise NH_3 flow to 0.5 cfm and cool solution to 70°C ± 2°C.

   (l) Sample solution with cooling water on the sampler jacket. Check the pH of the solution periodically until a pH of 8.5 to 9.0 is reached. Record final pH on data card.

   (m) When final pH is reached, immediately cut off NH_3 flow and cool solution to 20°C.

   (n) Determine the final precipitate volume and record on data card. If precipitate is greater than 5.0 gallons, check supervision. If ____ REl A and ____ REl B samples are requested, take at this point.

   (o) NOTE: As soon as solution reaches 30°C, proceed immediately to centrifugation step. Do not allow the slurry to remain in the precipitator any length of time, due to the formation of carbonates which will precipitate strontium.

3. Centrifugation of Rare Earth Precipitate

(a) Adjust the steam pressure supplying the control jet to the centrifuge to give 1.0 - 1.3 gallons per minute.

(b) Check that hold-up tank is empty and that all service valves are closed.

(c) Check that all valves servicing the centrifuge are closed.

(d) Set centrifuge skimmer in the "out" position.

(e) Turn on the D.C. brake switch.

(f) Turn on the oil pump to centrifuge bearings. Check the centrifuge oil supply tank level.

(g) Turn on centrifuge contact microphone.

(h) Turn on centrifuge motor.

(i) When tachometer reaches 1750 RPM, turn on the control jet from precipitator to centrifuge and check the following every 15 minutes:

1. Oil supply level.
2. Oil pump operating.
3. Centrifuge motor temperature.
4. Control jet flow rate.

(j) When the precipitator is empty, turn off the control jet to the centrifuge.

(k) Turn off the precipitator agitator jacket water.

(l) With centrifuge running, slowly skim to Notch 7. Skimming should be done over a period of 5 minutes.

(m) When skimming is finished, place skimmer arm in "out" position.

(n) Add 5.0 gallons of cold water to centrifuge.

(o) Slowly skim the centrifuge to Notch 7. Skimming should be done over a period of 5 minutes.

(p) When skimming is finished, place skimmer arm in "out" position.

(q) Turn off centrifuge and record the number of batches collected in the bowl.

4. Rare Earths Precipitate Dissolving

(a) Add 6.0 gallons of 5.0 N HNO_3 (nitric acid) to centrifuge. (Make-up: Add 2.0 gallons of 60 percent HNO_3 to centrifuge. Then add 4.0 gallons of water.)

(b) Turn on the steam to centrifuge coil by setting the steam controller to 100 lbs./hr. of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 30 minutes for 4 hours and check the following. (In case of split batch, dissolve the first part for one hour.)

1. Steam rate.
2. Solution temperature.
3. Centrifuge motor temperature.

(f) Turn off the steam to centrifuge by setting the steam controller to 0 lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil.

(i) Check that designated fraction tank is ready to receive solution.

(j) Jet solution to fraction tank, F-1021.

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

(n) Repeat Steps (k) through (m).

APPENDIX NO. 5-h

STRONTIUM PRECIPITATION PROCEDURE

Carbonate Precipitation of Strontium

1. Chemical Make-Up

   (a) 6.0 gallons 19 M NaOH (sodium hydroxide)

   (b) 7.0 lbs. Na_2CO_3 (sodium carbonate) dissolved in 6.0 liters of water

   (c) 6.0 gallons 1.0 N HNO_3 for dissolving precipitate

2. Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Jet RE-2 centrifugate from hold-up tank to Sr-2 precipitator.

   (d) Turn on contact microphone, agitator and process water to jacket.

   (e) With agitator off, record volume on data card.

   (f) Turn on precipitator agitator and sample. Code Sr-2 .

   (g) Add 6.0 gallons of 19 M NaOH to the precipitator.

   (h) Heat solution to 90°C ± 2°C and digest for 1.0 hour.

   (i) When digestion is completed, check pH of solution. Final pH should be 12.0 or greater. Add 19 M NaOH as necessary to reach pH of 12.0. Record final pH.

   (j) Add 7.0 pounds Na_2CO_3 dissolved in 6.0 liters of water. Flush solution addition line with one liter water.

   (k) Digest solution at 90°C to 95°C for 15 minutes.

   (l) Cool solution to 30°C.

   (m) If _____ Srl A or _____ Srl B samples are requested, take at this time. Record precipitate volume on data card.

3. Centrifugation of Strontium Precipitate

   (a) Adjust the steam pressure supplying the control jet to the centrifuge to give 1.0 to 1.3 gallons per minute.

(b) Check that hold-up tank is empty and that all service valves are closed.

(c) Check that all valves servicing the centrifuge are closed.

(d) Set centrifuge skimmer in the "out" position.

(e) Turn on the D.C. brake switch.

(f) Turn on the oil pump to centrifuge bearings. Check the centrifuge oil supply tank level.

(g) Turn on centrifuge contact microphone.

(h) Turn on centrifuge motor.

(i) When the tachometer reaches 1750 RPM, turn on the control jet from the precipitator to the centrifuge and check the following every 15 minutes:

1. Oil pump operating
2. Oil supply level
3. Centrifuge motor temperature
4. Control jet flow rate
5. Check hold-up tank volume.
   Keep volume under 250 gallons.

(j) When the precipitator is empty, turn off the control jet to the centrifuge.

(k) Turn off the precipitator agitator and jacket water.

(l) With the centrifuge running, slowly skim to Notch 7 on the centrifuge. Skimming should be done over a period of five minutes.

(m) When skimming is finished, place the skimmer arm in "out" position.

(n) Continue running centrifuge until the washes are completed.

(o) Add five gallons of water to the centrifuge.

(p) Five minutes after water addition, <u>slowly</u> skim to Notch 7 on centrifuge. Skimming should be done over a period of <u>five</u> minutes.

(q) When skimming is finished, place skimmer arm in the "out" position.

(r) Record the number of batches collected in centrifuge bowl and turn off centrifuge.

4. Strontium Precipitate Dissolving

(a) Add 6.0 gallons of 1.0 N HNO_3 (nitric acid) to centrifuge very slowly. (Make-Up: Add 5.0 gallons of water, then add 0.5 gallons of 60% HNO_3 to centrifuge. Flush line with two liters water.

(b) Turn on the steam to centrifuge coil by setting the steam controller to 100 lbs./hr of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 15 minutes for one hour and check the following:

1. Steam rate
2. Solution temperature
3. Centrifuge motor temperature

(f) Turn off the steam to centrifuge by setting the steam controller to 0 lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil.

(i) Check that designated fraction tank is ready to receive solution.

(j) Jet solution to designated fraction tank (F-421).

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

(n) Repeat Steps (k) through (m).

APPENDIX NO. 5-i

TECHNETIUM PRECIPITATION PROCEDURE

Magnetite Precipitation of Technetium

1. Chemical Make-Up

   (a) 5.0 gallons 60% HNO_3 (nitric)

   (b) 1.0 pound $Fe(NO_3)_3 \cdot 6\ H_2O$ (ferric nitrate) dissolved in 2.0 liters hot water

   (c) 12.0 liters 42.5% hydrazine

   (d) 5.0 gallons 19 M NaOH (sodium hydroxide)

   (e) 6 gallons 8 N HNO_3 for dissolving precipitate.

2. Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Jet solution from hold-up tank to precipitator.

   (d) Turn on contact microphone and agitator.

   (e) Heat solution to 60°C.

   NOTE: The following step will require close observation of the off gas due to the liberation of CO_2 gas.

   (f) Very carefully add 5.0 gallons of 60% HNO_3 to the precipitator while holding the temperature at 60°C and the off gas between 6 to 8 inches.

   (g) Final solution should be 0.3 N acid. Check for acid pH. If > 1.0, add one gallon shots of HNO_3 until pH is < 1.0.

   (h) Record precipitator volume with the agitator off.

   (i) Turn on the agitator and sample the solution. Code Tc2.

   (j) Add 1.0 pound $Fe(NO_3)_3 \cdot 6\ H_2O$ (ferric nitrate) dissolved in two liters hot water. Flush solution addition line with two liters water.

   (k) Add 6.0 liters of 42.5% hydrazine. Flush solution addition line with two liters water.

(l) Digest at 80°C for 15 minutes.

(m) Take a 10 milliliter sample for iron precipitation test to determine that all the iron has been reduced. Note typical magnetite color.

NOTE: Watch off gas closely on the following step.

(n) Add 5.0 gallons of 19 M NaOH (caustic) to precipitator. Add the first 2.0 gallons slowly over a period of ten minutes. Add the remainder of the 19 M NaOH (caustic) more rapidly over a period of five minutes. Check for pH of 12.0.

(o) Digest the solution for 45 minutes at 90°C.

(p) Cool solution to 70°C.

(q) Add 6 liters 42.5% hydrazine to precipitator. Flush solution addition line with two liters of water.

(r) Digest at 70°C for 15 minutes.

(s) Cool solution to 30°C and sample for precipitate volume. If _____A and _____B samples are requested, take at this time.

3. Magnetite-Technetium Precipitate Centrifugation

(a) Check that hold-up tank is empty and that all service valves are closed.

(b) Check that all valves servicing the centrifuge are closed.

(c) Set centrifuge skimmer in the "out" position.

(d) Turn on the D.C. brake switch.

(e) Turn on the oil pump to centrifuge bearings. Check the centrifuge oil supply tank level.

(f) Turn on centrifuge contact microphone.

(g) Turn on centrifuge motor.

(h) When the tachometer reaches 1750 RPM, turn on the control jet from the precipitator to the centrifuge and check the following every 15 minutes:

1. Oil pump operation
2. Oil supply level
3. Centrifuge motor temperature
4. Control jet rate, G.P.M.

(i) When the precipitator is empty, turn off the control jet.

(j) Turn off the agitator and jacket water.

(k) Slowly skim to Notch 7 on the centrifuge. Skimming should be done over a period of 5 minutes.

(l) When skimming is finished, place the skimmer arm in "out" position.

(m) Continue running centrifuge until the water washes are completed.

(n) Add five gallons of water to centrifuge.

(o) Five minutes after water addition, slowly skim to Notch 7 on centrifuge. Skimming should be done over a period of 5 minutes.

(p) When skimming is finished, place skimmer arm in "out" position.

(q) Turn off centrifuge.

4. Magnetite-Technetium Precipitate Dissolving

(a) Add 6.0 gallons of 8.0 N HNO_3 (nitric acid) to centrifuge. (Make-up: Add 2.5 gallons of water, then add 3.5 gallons of 60% HNO_3 very slowly to centrifuge.)

(b) Turn on the steam to centrifuge coil by setting the steam controller to 100 lbs./hr of steam and heat to 85°C.

(c) Hold temperature between 80°C and 85°C.

(d) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(e) Repeat the jogging procedure every 30 minutes for four hours and check the following:

1. Steam rate
2. Solution temperature
3. Centrifuge motor temperature

(f) Turn off the steam to the centrifuge coil by setting the steam controller to 0 lbs./hr.

(g) Turn on the process water to centrifuge coil.

(h) Cool solution to 60°C, then turn off the water to coil.

(i) Check that F-821 is ready to receive solution.

(j) Jet solution to designated fraction tank.

(k) Add 3.0 gallons of water to centrifuge.

(l) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(m) Jet solution to designated fraction tank.

(n) Repeat Steps (k) through (m).

5. Concentrate Waste Disposal

(a) Jet H-34 centrifugate to P-45 and sample solution. Code____Tc2W.

(b) Jet P-45 to S-324 to W-5.

APPENDIX NO. 5-j

OXALATE PRECIPITATION PROCEDURE - OX1

Oxalate Precipitation of Rare Earths

1. Chemical Make-Up

   (a) Approximately 15 gallons of 19 M C.P. NaOH. (Titrate to 0.1 N acid.)

   (b) Approximately 30 pounds oxalic acid. (Final solution to be 0.15 M oxalic.)

   (c) 5 pounds $NaNO_3$ added to oxalic acid.

   (d) 6.5 gallons 70% HNO_3.

   (e) 80 milliliters of 80% $Mn(NO_3)_2$.

2. Operation

   (a) Check that all jet and service valves to precipitator are closed.

   (b) Check that precipitator is empty and has 6 to 8 inches of off gas.

   (c) Jet 100 gallons of solution from F-23 to precipitator P-34.

   (d) Record precipitator volume with the agitator off.

   (e) Turn on the agitator and sample the solution. Code - Batch No. Oxalate No. (_____Ox1) .

   (f) Turn on contact microphone and agitator.

   (g) Adjust solution temperature to 50°C.

   (h) If pH meter is not operating due to high radiation, titrate all samples with 1.0 M NaOH.

   (i) Very carefully add 7.0 gallons of 19 M C.P. NaOH to the precipitator while holding the temperature at 60°C and the off gas between 6 and 8 inches. Titrate solution to 0.5 M.

   (j) Heat solution to 80°C.

   (k) Add 10 gallons oxalic acid - sodium nitrate solution.

   Make-Up

   1. Heat 6 gallons water to 80°C.

   2. Add 5 pounds $NaNO_3$ to water.

(k) (Cont'd)

3. Add 30 pounds oxalic acid to solution.

4. Build solution volume to ten gallons.

5. Immediately add solution to precipitator.

(l) Immediately cool precipitator to 25°C.

(m) Add 5.0 gallons 19 M C.P. NaOH.

(n) Digest at 25°C for one hour. Titrate solution during digestion as follows:

> Take 10 milliliters of slurry. Centrifuge. Decant to clean tube. Add 1.0 M NaOH, 1.0 milliliter at a time until percent precipitate increases. Calculate normality. Adjust to 0.1 N free acid by adding one gallon of 19 M NaOH for each 0.1 N increment.

(o) Sample for precipitate volume and if A and B samples are requested, take at this time.

3. Rare Earth Oxalate Precipitate Centrifugation

(a) Check that hold-up tank is empty and that all service valves are closed.

(b) Check that all valves servicing the centrifuge are closed.

(c) Set centrifuge skimmer in the "out" position.

(d) Turn on the D.C. brake switch.

(e) Turn on the oil pump to centrifuge bearings. Check the centrifuge oil supply tank level.

(f) Turn on centrifuge contact microphone.

(g) Turn on centrifuge motor.

(h) When the tachometer reaches 1750 RPM, turn on the control jet at 1.0 G.P.M. from the precipitator to the centrifuge and check the following every 30 minutes:

1. Oil pump operation
2. Oil supply level
3. Centrifuge motor temperature
4. Control jet rate, G.P.M.

(i) When the liquid level reaches 10%, turn off agitator.

(j) When the precipitator is empty, turn off the control jet.

(k) Turn off the agitator, jacket water and reflux condenser.

(l) Slowly skim to Notch 8 on the centrifuge. Skimming should be done over a period of five minutes.

(m) When skimming is finished, place the skimmer arm in "out" position.

(n) Continue running centrifuge until the water washes are completed.

(o) Add five gallons of water to precipitator and jet to centrifuge.

(p) Five minutes after water addition, slowly skim to Notch 8 on centrifuge. Skimming should be done over a period of five minutes. Repeat Step (o) once. After last H_2O wash, skim close to cake. Leave a minimum of three gallons in centrifuge bowl.

(q) When skimming is finished, place skimmer arm in "out" position.

(r) Turn off centrifuge. Transfer supernate from "H" tank to "P" tank. Sample as _____OX1W and hold for analysis.

4. Rare Earth Oxalate Precipitate Dissolving

(a) Add 80 milliliters of 80% $Mn(NO_3)_2$ to the centrifuge bowl.

(b) Add 6.0 gallons of 14.0 N HNO_3 to centrifuge.

(c) Turn on the steam to centrifuge coil by setting the steam controller to 100 lbs./hr of steam and heat to 90°C.

(d) Hold temperature between 85°C and 90°C.

(e) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(f) Repeat the jogging procedure every 30 minutes for four hours and check the following:

1. Steam rate
2. Solution temperature
3. Centrifuge motor temperature

(g) Turn off the steam to the centrifuge coil by setting the steam controller to 0 lbs./hr.

(h) Turn on the process water to centrifuge coil.

(i) Cool solution to 20°C, then turn off the water to coil.

(j) Check that H-89, Cell 9, is ready to receive solution.

(k) Jet solution to H-89.

(l) Add 3.0 gallons of water to centrifuge.

(m) Turn on D.C. brake switch and jog centrifuge to 1750 RPM, then turn it off.

(n) Jet solution to H-89.

(o) Repeat Steps (l) through (n).

APPENDIX NO. 5-k

YTTRIUM EXTRACTION PROCEDURE

A. Yttrium Extraction

I. Chemical Make-Up

1. 3 gallons of 19 M NaOH (C.P. Grade)
2. 26.4 liters of Amsco or Super Sol
3. 6.6 liters of D2EHPA
4. 3.0 gallons 1 N HNO_3

II. Operation

1. Check that all jets and valves are closed on Tank H-89 and tank is ready to receive solution from G-34. Jet the acid solution from G-34 to H-89.
2. Follow with a 5-gallon water rinse through G-34 to H-89.
3. Again add a 5.0 gallon water rinse to G-34 and jet to H-89.
4. Check that all jets and valves are closed on Tank N-19 and tank is ready to receive solution from H-89. Jet solution from H-89 to N-19.
5. Cool N-19 to 30°C.
6. Turn off agitator and record volume on data card. Assume specific gravity of 1.1.
7. Turn on agitator and sample. Code_____Y1.
8. 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.
9. Agitate N-19 for 10 minutes and sample solution for acid normality. Make titration in Cell 19. Record acid normality on batch data card.
10. Adjust acid normality to 1 N $\pm$ 0.1 free acid by adding 500 ml of 19 M C.P. NaOH or 500 ml C.P. HNO_3 for each 0.1 above or below 1.0 N. Hold temperature at 20°C to 25°C with water on jacket. Flush addition line with 2.0 liters of water after each NaOH addition.
11. Add 6.6 liters of D2EHPA to N-19.

12. Add 26.4 liters of Amsco to N-19.

13. Agitate N-19 for 10 minutes at 20 to 25°C.

14. Turn off agitator and let settle for 10 minutes.

15. Check R-79 that all jets are off and tank has been rinsed and is ready to receive solution from N-19.

16. Open valve from N-19 to R-79 and separate aqueous phase from the organic by air lift flow of aqueous to R-79. Aqueous phase contains all RE except yttrium. Regulate air flow to 37.5% on air lift to give 3 gallons per minute.

    NOTE: When the organic phase reaches the short leg on interface, the alarm will sound and valve from N-19 to R-79 will automatically close.

17. When the alarm sounds, cut off hand switch.

18. Check N-29 that all valves and jets are closed and tank is empty.

19. Jet the aqueous solution containing cerium and promethium from R-79 to N-29.

20. Add 1.5 gallon portion of 1 N HNO_3 to N-19. Settle for 3 minutes.

21. Open valve from N-19 to R-79 and separate aqueous phase from the organic by regulating air flow to 37.5% on air lift to give a three gallon per minute flow.

    NOTE: When the organic phase reaches the short leg on interface, the alarm will sound and the valve from N-19 to R-79 will automatically close.

22. When the alarm sounds, cut off hand switch.

23. Check N-29 that all valves and jets are closed and tank is ready to receive the HNO_3 rinse.

24. Jet the aqueous solution from R-79 to N-29.

25. Add 1.5 gallons of 1.0 N HNO_3 to N-19 and let it settle for three minutes.

26. Open valve from N-19 to R-79 and separate aqueous phase from the organic by regulating air flow to 37.5% on air lift to give a three gallon per minute flow.

    NOTE: When the organic phase reaches the short leg on interface, the alarm will sound and valve from N-19 to R-79 will automatically close.

27. When the alarm sounds, cut off hand switch.

28. Check that N-29 is ready to receive the HNO_3 rinse.

29. 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: Step B. 1 below can be done during the volume reduction step in N-29.

B. Removal of Organic Phase

1. Check R-29 that all valves and jets are off and tank is ready to receive organic Y^{91} waste from N-19.

2. Transfer the organic waste from N-19 to R-29. Regulate air flow to 50% on air lift.

3. Add 35 gallons H_2O to N-19 and agitate 5 minutes and transfer to R-29. Regulate air flow to 50% on air lift.

   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 pump when liquid level reaches 5% by volume.

4. Pump the organic Y^{91} waste from R-29 to W-6. Check with supervisor.

5. Follow organic with 50 gallons water wash to W-6.

6. Continue with Ce^{144} Extraction Procedure.

APPENDIX No. 5-1

CERIUM EXTRACTION PROCEDURE

A. Ce^{144} Extraction

I. Chemical Make-Up

1. 3 gallons of 19 M NaOH (C.P. grade)
2. 10 liters of 1.0 M $KMnO_4$ (add 1580 grams of $KMnO_4$ and 60 ml of 70% HNO_3 to 10 liters of hot water, 65°C)
3. 16 gallons 2 N HNO_3
4. 33.0 liters Amsco
5. 9.9 liters Amsco
6. 16.5 liters Amsco
7. 6.6 liters D2EHPA
8. 10 liters 1 M citric acid
9. 30 gallons 1 N HNO_3 (dilute 15 gallons of 2 N HNO_3 with 15 gallons water)
10. 26.4 gallons 10 N HNO_3
11. 3 liters 30% H_2O_2

II. 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.

   NOTE: If more than 8 hours time has 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 time has elapsed, continue with next step.

7. Adjust acid normality to 1 N ± 0.05 free acid. Add 250 ml of acid or base for each 0.05 change in normality. Rinse addition line

with two liters H_2O. Hold temperature at 20 to 25°C with water on jacket.

8. Add 1.0 N HNO_3 to bring volume up to 100 liters.

   NOTE: Final volume of aqueous in N-29 should be 100 liters or 26.4 gallons of 1 N free acid.

9. Turn off agitator and record volume of aqueous solution in N-29 on data card. Assume a specific gravity of 1.1.

10. Make up 10 liters of 1 M $KMnO_4$. Add 1580 grams of $KMnO_4$ to 10 liters of hot water. Acidify KMnO4 solution with 60 ml of 70% HNO_3.

    NOTE: Operations have to proceed rapidly from step to step from this step on.

11. Turn on agitator and jacket water on N-29.

12. Add 5 liters of 1 M $KMnO_4$ to N-29.

13. Add 5 liters of 2.0 N HNO_3 solution to N-29.

14. Add 6.6 liters of D2EHPA to N-29.

15. Add 9.9 liters of Amsco to N-29.

16. Agitate for 15 minutes from time of last Amsco addition.

    NOTE: Sample N-29 every 5 minutes before Step 17. 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 card the total volume of extra 1 M $KMnO_4$ added. Wash each liter $KMnO_4$ with 1.0 liter 2 N HNO_3.

17. At end of digestion period, immediately add 16.5 liters of Amsco and agitate 15 minutes from time of last Amsco addition. Also see above note in regard to maintaining color.

18. 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.)

    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.

19. Agitate 10 minutes after addition of citric acid.

20. Turn off agitator and let settle for 10 minutes.

21. Check R-79 that all jets are off and tank is ready to receive solution from N-29.

22. Open valve from N-29 to R-79 and separate aqueous phase from the organic by air lift flow of aqueous to R-79. Regulate air flow to 37.5% to air lift to give about 3 gallons/minute.

    NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and valve from N-29 to R-79 will automatically close.

23. When the alarm sounds, cut off hand switch.

24. Check N-19 that all valves and jets are closed and tank is empty and ready to receive solution from R-79.

25. Jet the aqueous solution containing promethium from R-79 to N-19 and hold.

26. Add 1.5 gallons of 1.0 N HNO_3 to N-29 and settle 3 minutes.

27. Open valve from N-29 to R-79 and separate aqueous phase from the organic by air lift flow of aqueous to R-79. Regulate air flow to 37.5% to air lift 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 valve from N-29 to R-79 will automatically close.

28. When alarm sounds, cut off hand switch.

29. Jet the aqueous solution from R-79 to N-19 and hold.

30. Add 1.5 gallons of 1.0 N HNO_3 to N-29 and settle three minutes.

31. Open valve from N-29 to R-79 and separate aqueous phase from the organic by air lift flow of aqueous to R-79. Regulate air flow to 37.5% to air lift 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 valve from N-29 to R-79 will automatically close.

32. When the alarm sounds, cut off hand switch.

33. Jet the aqueous solution from R-79 to N-19 and hold for the Silica Manganese and Nickel Removal Procedure.

B. Acid Rinse of Organic Phase

1. Add 26.5 gallons of 10 N HNO_3 to N-29.

2. Agitate solution in N-29 for 10 minutes at 25°C.

3. Cut off agitator and let 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. Regulate air flow to 37.5% to air lift to give ~ 3 gallons/minute.

   NOTE: When the organic phase reaches the short leg on interface instrument, the alarm will sound and valve from N-29 to R-79 will automatically close. Also close hand switch.

6. Check R-39 that all jets and valves are closed and tank is empty.

7. Jet acid rinse from R-79 to R-39.

8. Add 2.5 gallons of 1 N HNO_3 to N-29.

9. Open valve from N-29 to R-79 and separate aqueous phase from organic. Regulate air flow at 37.5% to air lift to give a flow of 3 gallons/minute.

   NOTE: When the organic phase reaches the short leg on interface instrument, the alarm will sound and valve from N-29 to R-79 will automatically close. When the alarm sounds, cut off the hand switch.

10. Jet the acid rinse from R-79 to R-39.

11. Add 2.5 gallons of 1.0 N HNO_3 to N-29.

12. Open valve from N-29 to R-79 and separate aqueous phase from organic. Regulate air flow at 37.5% to air lift to give a flow of 3 gallons/minute.

    NOTE: When the organic phase reaches the short leg on interface instrument, the alarm will sound and valve from N-29 to R-79 will automatically close. When the alarm sounds, cut off the hand switch.

13. Jet the acid rinse from R-79 to R-39 and agitate.

14. Sample R-39 for the lab. Code ____CelW.

15. Cut off R-39 agitator and then record R-39 volume on the batch data card and assume a specific gravity of 1.1.

16. Jet R-39 solution to W-6.

C. Re-Extract Ce^{144} from Organic Phase

1. Add 33.0 liters of Amsco to N-29.

2. Add 8 gallons of 2 N HNO_3 to N-29. Read Step 3.

3. Add 3 liters H_2O_2 concurrently with the acid solution.

4. Agitate N-29 for 10 minutes at 25°C.

5. Cut off agitator and let settle for 10 minutes.

6. Check R-79 that all jets are off and tank is empty and ready to receive the Ce^{144} fraction.

7. Open valve from N-29 to R-79 and separate the aqueous from organic by air lift to R-79. Regulate air flow to 37.5% to give ~ 3 gallons/minute.

   NOTE: When the organic reaches the interface probe, the alarm will sound and valve from N-29 to R-79 will close automatically. Also 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 Ce^{144} solution from R-79.

10. Jet R-79 to H-511.

11. Add 1.5 gallons of 1 N HNO_3 to N-29.

12. Open valve from N-29 to R-79 and separate the aqueous from organic by air lift to R-79. Regulate air flow to 37.5% to give a 3-gallon-per-minute flow.

    NOTE: When the organic reaches the interface probe, the alarm will sound and valve from N-29 to R-79 will close automatically. Also close the hand switch when the alarm sounds.

13. Jet R-79 to H-511.

14. Add 1.5 gallons of 1 N HNO_3 to N-29.

15. Open valve from N-29 to R-79 and separate the aqueous from organic by air lift to R-79. Regulate air flow to 37.5% to give a 3-gallon-per-minute flow.

    NOTE: When the organic reaches the interface probe, the alarm will sound and valve from N-29 to R-79 will close automatically. Also close the hand switch.

16. Jet R-79 to H-511 which is now ready for Cell 11 Procedure.

17. Open valve from N-29 to R-29 and transfer to organic waste to R-29 by regulating the air flow to 50% on air lift.

18. Add 36 gallons H_2O to N-29 and agitate 5 minutes.

19. Transfer solution from N-29 to R-29.

19. <u>NOTE</u>: 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 pump when liquid level reaches 5% by volume. Cut off starter switch.

20. Pump the organic waste from R-29 to W-6. Check with supervisor.

21. Follow the organic with a 50-gallon water wash.

APPENDIX NO. 5-m

SILICA, MANGANESE AND NICKEL REMOVAL PROCEDURE

I. Chemical Make-Up

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. Add 2 ml of 16 N HNO_3 to 4 liters of water.
5. 133 liters of 2 N HNO_3
6. 33 liters of Amsco.

II. Operation

A. Promethium Extraction of Feed

1. Reduce volume in N-19 to 80 liters by heating to 95°C.
2. When volume in N-19 is 80 liters, cool to 25°C.
3. Add 4.0 liters of 19.0 M C.P. NaOH to N-19 and follow with a 2-liter H_2O rinse.
4. Titrate sample for acid normality.
5. Adjust acid normality to 0.1 N by adding 500 ml of 19 M NaOH or 500 ml of 16 N HNO_3 for each 0.1 N to be raised or lowered and follow each addition with a 2.0 liter H_2O wash.
6. Adjust pH to 1.6 to 2.3 by adding 125 ml 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 card.
8. Add 13.2 liters of D2EHPA.
9. Add 19.8 liters of Amsco.
10. Agitate for ten minutes.

    NOTE: 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 supervisor.

11. Turn off agitator and settle for ten minutes.

12. Take sample in Cell 19.

    NOTE: 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.

13. Check pH of solution.

14. Adjust pH to 1.6 to 2.3 by adding 125 ml increments of 19 M C.P. NaOH; follow each with a 2.0 liter water rinse.

15. Agitate N-19 for ten minutes.

16. Let settle for ten minutes.

17. Take sample in Cell 19.

    NOTE: 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.

18. Check pH of solution.

19. Adjust pH to 1.6 to 2.3 by adding 125 ml increments of C.P. 19 M NaOH until the pH remains at 1.6 to 2.3. Follow each NaOH addition with 2.0 liter water rinse.

20. When pH is in range of 1.6 to 2.3, continue with procedure.

21. Check R-79 to be sure that all jets are off and tank is ready to receive solution from N-19.

    NOTE: Proceed only after a ten minute settling period in N-19 after the last sample has been taken.

22. Open valve from N-19 to R-79 and separate aqueous phase from the organic by air lift flow of aqueous to R-79. Regulate air flow to 37.5% to air lift to give about three gallons per minute of solution.

    NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and valve from N-19 to R-79 will close automatically.

23. When the alarm sounds, cut off hand switch.

24. Jet solution from R-79 to R-39.

    NOTE: At this time the feed material in R-39 has the promethium product removed from it and the organic solution contains the promethium product.

25. Sample solution in R-39 for the lab. Code_____SilW.

26. Jet R-39 solution to W-6.

27. Add 50 liters of 2 N nitric acid to N-19.

28. Add 33 liters of Amsco to N-19.

29. Agitate N-19 for 15 minutes.

30. Turn off agitator and settle for ten minutes.

31. Check R-79 that all jets are off and tank is ready for solution from N-19.

32. Open valve from N-19 to R-79 and separate aqueous phase from the organic by air lift flow of aqueous to R-79. Regulate air flow to 37.5% to air lift to give about three gallons per minute flow of solution.

    NOTE: When the organic phase reaches the short leg on the interface, the alarm will sound and valve from N-19 to R-79 will close automatically. When alarm sounds, cut off hand switch.

33. Check that all valves to F-2 and P-89 are closed and P-89 has off gas.

34. Pressurize the bottom of F-2 filter with air regulator at 5 psi and valve 10933 open.

35. Jet R-79 to P-89.

36. Add 50 liters of 2 N nitric acid to N-19.

37. Add 33 liters of Amsco to N-19.

38. Agitate N-19 for 15 minutes.

39. Turn off agitator and settle for ten minutes.

40. Check R-79 that all jets are off and tank is ready for solution from N-19.

41. Open valve from N-19 to R-79 and separate aqueous phase from the organic by air flow of aqueous to R-79. Regulate air flow to 37.5% to air lift to give about three gallons per minute flow of solution.

    NOTE: When the organic phase reaches short leg on interface, the alarm will sound and valve from N-19 to R-79 will close automatically.

42. When the alarm sounds, cut off hand switch.

43. Jet R-79 solution to P-89 which is now ready for the Promethium Oxalate Procedure.

B. Organic Removal Procedure

1. Open valve from N-19 to R-29 and transfer the organic waste to R-29 by regulating the air flow to 50% on the air lift.

2. Add 36 gallons of water to N-19 and agitate five minutes and then transfer to R-29. R-29 should have a liquid level of 40% by volume to prime U-91 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 R-29 to W-6. Check with supervisor.

4. Follow organic with 50-gallon water wash.

APPENDIX NO. 5-n

PROMETHIUM PRECIPITATION PROCEDURE

A. Pm Ox Precipitation Procedure in P-89

I. Chemical Make-Up

1. 3.0 gallons 19 M NaOH (C.P. grade)
2. 4 liters 1.8 M oxalic acid (one-half pound per liter)
3. 4 liters of 7 N HNO_3
4. 3 milliliters 80% $Mn(NO_3)_2 \cdot 6H_2O$

II. Operation

1. Agitate solution in P-89 for 5 minutes and record volume with agitator off.
2. Sample P-89 for the lab. Code_____OX2.
3. Turn on P-89 agitator and adjust the solution to 0.25 to 0.30 N free acid by adding 500 ml of 16 N HNO_3 or 500 ml 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 off gas between 6 to 8 inches.
4. Heat P-89 to 80°C.

   NOTE: Read Steps 5 and 6 before proceeding with 5. (Make-up = two pounds of oxalic acid in 4 liters of hot water, 80°C).

5. Add 2 liters of oxalic acid to P-89. Follow oxalic acid with 2 liters H_2O.
6. Cool P-89 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.

Test for Complete Precipitation

Take a ten milliliter sample and centrifuge. Pour the supernate into a clean centrifuge tube. Add one milliliter of saturated oxalic acid solution and stir. Centrifuge. If no precipitate is formed, the rare earth oxalate precipitation is complete. 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. Only the promethium oxalate appears green in the cell and is an indication of an incomplete precipitation. If the precipitation is not complete, add two

6. (Cont'd)

liters of oxalic acid to P-89. (Make up one pound of oxalic acid in two liters of hot water.) Digest for one hour and then repeat test.

7. Cut off air pressure to F-2 by closing valve 10933 and set air regulator to 5 psi.

8. Check that R-39 is empty and is ready to receive the filtrate from F-2.

9. Start vacuum jet and open valve from F-2 to vacuum jet. Open valve from P-89 to F-2.

10. Filter out the Pm Ox precipitate in F-2.

11. Cut off P-89 agitator when liquid level reading is approximately 20% by volume.

12. Record filtration rate.

NOTE: Volume of R-39 during filtration. The jet dilution may overfill tank.

NOTE: Continue vacuum jet according to instructions from supervision. This will be determined on filtration rate. F-2 has approximately 2-3/4 gallons hold-up.

13. After filtration is finished, rinse P-89 with water spray, two 2-minute portions.

NOTE: Flow rate on P-89 water spray is approximately 0.8 gallons per minute.

14. Continue vacuum jet for calculated time on each of the water rinses from P-89.

NOTE: It is essential that no liquid remain in F-2 before Step 15. Let vacuum jet run 15 minutes or 20% of total filtration time whichever is the longer after F-2 should be dry.

15. Cut off vacuum jet to R-39 and close valve from F-2 to vacuum jet. Also close valve from P-89 to F-2.

16. Record R-39 volume and sample filtrate in R-39. Code_____OX2W.

17. Jet R-39 solution to W-6 tank.

18. Add 50 gallons of water to R-39, then jet R-39 to W-6.

B. Pm Ox Precipitate Dissolving in F-2

1. Check all valves to and from F-2 to see that they are closed.

2. Open off-gas valve from F-2 to P-89, off gas condenser H_2O and P-89 jacket water.

   NOTE: Do not close off-gas valve during dissolving of Pm Ox in F-2.

3. Pressurize the bottom of F-2 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 F-2 during dissolving of precipitate. This is to prevent acid from leaking through filter.

4. Add 5 gallons of 16 N HNO_3 with 15 milliliters of 80% $Mn(NO_3)_2 \cdot 6 H_2O$ to P-89. Open valve from P-89 to F-2 to drain acid solution to F-2.

   NOTE: When sampling F-2, leave jet open only a minimum time to get sample.

5. Leave valve open from P-89 to F-2 after acid has drained to F-2.

6. Heat F-2 to 95°C and digest for four hours.

7. Cool F-2 to 25°C.

8. If requested, sample F-2. Solution Code_____PM1.

9. Record F-2 volume on data card. This volume can be obtained by reading P-89 liquid level and getting the volume from F-2 calibration.

10. Transfer product from F-2 to product carrier in Cell 15.

C. Product Transfer Procedure to Cell No. 15

1. Cap off long leg of product carrier.

   NOTE: 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.

2. Place promethium carrier in ten-ton dolly in Cell 15.

3. Connect Snaptite fitting on short leg of carrier from Cell 9.

4. Connect Snaptite fitting on short leg of carrier from Cell 15 to P-45, P-12 and E-16, jet No. J56-1.

5. Check that P-45 is empty and will not be in operation for approximately 3 to 4 hours.

6. Open valve in Cell 15 on suction side of Jet J56-1.

7. Open valve on discharge of J56-1 to P-45.

8. Close valve on air line to bottom of F-2 and set pressure regulator to 0.

9. Open valve from F-2 to Cell 15 on Panelboard 4.

10. Open manipulator operated valve in Cell 15 from F-2.

11. Turn on Jet J56-1.

12. Time for transfer may vary so check F-2 by sampling after product has been transferred to be sure filter vessel is empty.

13. Rinse F-2 with two 4-liter portions of 16 N C.P. HNO_3. Transfer each rinse to product carrier.

14. After rinses, disconnect line to vacuum jet J56-1 and flush with water to P-45.

15. Close valve to Jet J56-1 and manipulator operated valve in Cell 15.

16. Cut off steam jet No. J56-1 and close valve to P-45.

17. Close panelboard valve F-2 to Cell 15.

18. Turn off F-2 off gas condenser water.

19. After each transfer the tygon lines will be discarded but the Snaptites are to be decontaminated and put on new tygon. Tygon lines are six-feet long.

20. If the filtration through F-2 was slow, check with supervision for a filter clean-up procedure.

APPENDIX No. 5-o

CERIUM PURIFICATION PROCEDURE

I. Ce^{144} Oxalate Precipitation in P-511

A. Chemical Make-Up

1. 6 liters concentrated NH_4OH

   NOTE: Oxalic acid should be made up 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. Operation

NOTE: Check Cell 9 procedure on re-extraction of Ce^{144} from the organic phase and Ce^{144} transfer to Cell 11.

NOTE: Do not exceed three batches of Ce^{144} in Cell 11 at one time.

1. Transfer cerium product from R-79 to H-511 and record volume.

   L.L. _____ %
   Assume Sp.Gr. 1.1
   Volume _____

2. Check that P-511 is empty and all valves and services are closed.

   NOTE: V-14, V-18 and V-12 valves are operated with manipulators. Also check that P-511 has six to eight inches of off gas.

   NOTE: Be sure Valves V-14 and V-18 are closed.

3. Connect filter to filter top by the following steps:

   (a) Check that filter is empty and ready to install.

   (b) Place filter on dolly.

   (c) Place "O" ring gasket on top of filter flange carefully.

   (d) Push dolly over pneumatic jack and lock in place.

   (e) Open V band clamp halves to fully open position.

   (f) Hook up Snaptite fitting to R-111 and push filter against stops on dolly. Close off gas valve to R-111 and open V-10 to vacuum jet.

   (g) Regulate air to pneumatic jack to 20 pounds psi and carefully raise filter into position.

(h) Use wrench to tighten V band securely.

(i) Leave jack in the "up" position.

(j) Add 50 liters H_2O to P-511 and open V-14 and check filter "O" ring gasket for leaks. If no leak is noted, filter the H_2O to R-111 and to drain.

(k) Check R-111 that all valves and services are closed except off gas and vessel is empty.

NOTE: Hand operated valves on R-111 are V-13 air sparge, V-16, V-17 on sample lines to Cell 19, to V-9 on vacuum header and V-10 to vacuum jet J101-11. The hand operated valves are located on cell face.

(l) Close off gas and open V-10 to vacuum jet. Turn on steam to vacuum jet for water transfer to R-111.

(m) When water has been transferred to R-111, close V-10 and cut off jet. Allow vacuum on R-111 to return to zero slowly to prevent suck-back to instrument cubicle.

(n) Open V-10 and drain water to waste.

(o) Close V-10 and open off gas valve.

4. Disconnect Snaptite fitting from R-111 and connect to air line. Close V-14 valve from P-511 to filter and turn on 5 pounds air to bottom of filter. This valve is located on panelboard.

5. Check pressure indicator on filter vessel that 5 pounds of air is maintained during CeOx precipitation. This is to prevent solution from leaking through Valve V-14.

6. Jet one batch of Ce^{144} product from H-511 to P-511 and turn on P-511 agitator.

NOTE: One batch of Ce^{144} product from R-79 should be ~ 13.5 gallons and should contain ~ 75 M of free HNO_3 acid, with jet dilution.

7. Add one gallon of water to H-511 and jet to P-511 if H-511 has only one batch.

8. Repeat Step 7.

9. Record P-511 volume with agitator off.

L.L. _____ %
Assume Sp. Gr. 1.1
Volume _____ Gallons

10. Turn on P-511 agitator

11. Sample P-511 in Cell 19 after ten minutes of agitation. Sample Code _____OX3. (Dilute 1 to 500 milliliters.)

12. Take 5 milliliters of original sample and titrate for acid normality with 1.0 N NaOH.

13. Heat P-511 to 50°C with agitation. Set steam regulator valve to P-511 jacket on panelboard and open steam outlet and inlet valves located on cell face.

14. Adjust acid normality to 0.2 normal ± 0.1 N free acid with concentrated ammonium hydroxide (NH_4OH), while holding temperature at 50°C and off gas between 6 to 8 inches.

15. The following additions of NH_4OH were calculated on a volume of 60 liters or 15.85 gallons of starting solution in P-511 and concentrated NH_4OH at 14 M.

    NOTE: Check normality after each addition.
    First addition 3 liters slowly - ~ 5 minutes
    Second addition 1 liter slowly - ~ 5 minutes
    Third addition 0.2 liter slowly - ~ 2 minutes

    NOTE: Solution addition funnel to P-511 is located on second level.

II. Oxalic Acid Precipitation of Ce^{144}

1. Heat solution in P-511 to 80°C with agitation. Set regulator on panelboard to 80°C and open steam inlet and outlet valves located on cell face.

   NOTE; Be sure oxalic acid is in solution, and that there is no delay in the procedure once the oxalic addition to P-511 has started.

2. At 80°C add 2 liters of 1.8 M oxalic acid to P-511. (Addition funnel located on second level.)

3. Cool P-511 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 P-511 too fast.

4. At 60°C add 4 liters of 1.8 M oxalic acid to P-511.

5. Cool P-511 to 40°C over a period of 15 minutes.

6. At 40°C add 4 liters of 1.8 M oxalic acid to P-511 and hold at 40°C for 10 minutes.

7. Cool P-511 to 25°C and leave water on until after filtration.

   NOTE: Check Step No. 1, Filter Operation, to prevent delay in filtration. If filtration is delayed add 1 liter of 1.8 M oxalic acid every 30 minutes, delayed up to two hours. Check with supervision.

8. Begin the filtration as soon as possible.

III. Filter Operation

1. Check R-111 that all valves and services are closed except off gas and vessel is empty.

   NOTE: Hand operated valves on R-111 are V-13 air sparge, V-16, V-17 on sample lines to Cell 19, V-9 to vacuum header and V-10 to vacuum jet J101-11. The hand operated valves are located on cell face.

2. Cut off air pressure to filter vessel.

3. Disconnect Snaptite from air line and connect to R-111.

4. Turn on steam to vacuum jet J101-11 from R-111 and close off gas valve.

5. Open V-10 to vacuum jet. Open V-14 manipulator valve from P-511 to filter vessel.

6. Filter out the CeOx precipitate.

7. When P-511 liquid level reaches 10% of tank capacity, turn off agitator.

   NOTE: Vacuum on R-111 should decrease when all solution has been filtered from P-511 to R-111.

8. After the filtration has finished, rinse P-511 with water spray, two 1-minute portions. (Open supply valve on face of cell and V-12 manipulator operated valve.)

   NOTE: Flow rate on P-511 water spray is ~ 4 gallons per minute.

9. Continue vacuum jet until a vacuum break on R-111 is noted for each water rinse.

10. After water rinse close valve from P-511 to filter (V-14) and water to P-511 jacket.

11. Close V-10 to vacuum jet and turn off steam to vacuum jet J101-11. When R-111 vacuum gauge returns to ~ 2 inches, open off-gas valve to R-111 on panelboard.

12. Disconnect Snaptite from filter to R-111 and cap off line to R-111 with blank Snaptite fitting.

13. Record R-111 volume

LLL. _____ %
Assume Sp.Gr. 1.1
Volume _____ Gallons

NOTE: Check that off-gas valve is open. Do not overcome off gas on Step No. 14.

14. Sparge R-111 with air sparger for 5 minutes. Valves are located on cell face.

15. Cut off air sparge to R-111.

16. Sample R-111 in Cell 19.

NOTE: Check V-16 and V-17 that valves are open before sampling. Valves are located on cell face.

17. Sample Code _____Ox3W.

18. Hold solution in R-111 for sample results.

19. If results on sample from R-111 show less than 15 millicuries per milliliter of Ce^{144}, drain solution to S-523 and close drain valve.

IV. Calcination of Precipitate

1. Disconnect V band on filter top and open.

2. Lower filter and remove "O" ring gasket. Pull dolly and filter to front of cell and check precipitate volume. Tilt filter on dolly to drain solution from filter bottom.

3. Replace filter and hang loosely on V band clamp.

4. Insert thermocouple into filter discharge tube.

5. Lower pneumatic jack and remove dolly. Place furnace around filter.

6. Turn on filter furnace and set controller at 670°C (maximum).

Time _____

7. After reaching 670°C allow to calcine for two hours.

Time at 670°C _____

8. After calcining for two hours, turn off furnace and allow to cool to 200°C.

Time Off _____
Time Temp. at 200°C _____

9. Open furnace and allow filter to cool to 100°C.

Time at 100°C _____

10. After temperature of furnace reaches 100°C, place dolly over locater ring and raise elevator.

11. Open V band and lower filter on dolly.

12. Remove thermocouple from filter discharge tube.

13. Pull dolly to approximately the center of table or a convenient position for removing the powder.

V. Powder Removal From Filter

1. Hook up cyclone Snaptite outlet to R-111 filter line.

   NOTE: Be sure cups are clamped tight on cyclone separator.

2. Check R-111 that all valves are closed, except V-10 to vacuum jet and tank is empty.

   NOTE: Do not allow water to be pulled into cyclone separator.

3. Turn on steam to R-111 vacuum jet J101-11.

4. Remove powder from filter into cyclone.

5. When powder transfer is completed, cut off vacuum jet. Close V-10 and open off gas to R-111.

6. Disconnect Snaptite connection from cyclone separator to R-111 and cap off line to R-111.

7. Vibrate the conical collector by tapping and carefully remove the powder vessels.

8. Place covers on vessels and transfer to Cell 12.

APPENDIX NO. 5-p

CESIUM PURIFICATION PROCEDURE

1. Chemical Make-Up Based on a 20,000 Curie Run

   (a) ~ 20 pounds ammonium alum

   (b) ~ 1.0 liter 70% HNO_3

   (c) ~ 2,250 ml 42.5% hydrazine hydrate

   (d) ~ 20 liters 12.0 N HCl

   (e) ~ 77 liters 1.5 M NaOH in the scrubber head tank (make-up = 6 liters of 19 M NaOH diluted to 77 liters)

   (f) ~ 500 grams of platinum

   (g) ~ 2,000 ml 30% H_2O_2

   1. HCl solutions to be added through the Area 33 funnel. Small quantities of HNO_3, hydrazine and hydrogen peroxide will be added at Cell 13.

2. Operational Checks

   (a) All stop cocks and pinch clamp valves are closed.

   (b) Transfer pump in operating condition and switch is off.

   (c) Drain the Cell 11 hot off gas scrubber.

   (d) Empty the Cell 13 vacuum scrubber. Then add 4.0 liters of 1.5 M NaOH to the vacuum scrubber.

   (e) Run 1.5 M NaOH from the head tank to Cell 13 hot off gas scrubber at 45% flow on rotometer. Continue until a change is requested.

   (f) Crystallizers have off gas by the off gas instrument on evaporator.

   (g) Crystallizers are empty except for crystal bed.

   (h) Portable dip legs removed from crystallizer.

   (i) Check that C-38 transfer line is in C-513 and locked in place.

3. Operations for C-38 to Cell 13 Transfer

   (a) Set up communications so that Cell 13 operator can be heard at control Cell 18 teletalk.

(b) Open the manual C-38 transfer valve.

(c) Notify the Panelboard 3 operator to start jetting from C-38 to Cell 13.

(d) Transfer 33 liters of solution from C-38 to Cell 13. Then immediately shut off the transfer jet and allow the transfer line to drain.

(e) Close the transfer line manual valve.

4. Crystallization Operations

(a) Turn on crystallizer agitator.

(b) Heat solution to 90°C.

(c) Digest solution at 90°C for 15 minutes.

(d) Turn on chilled water to crystallizer coil and cool solution to 20°C.

(e) Continue cooling and agitating solution for 15 minutes after crystals have formed.

(f) Turn off crystallizer agitator.

(g) Settle the crystal bed for 15 minutes. Record crystal volume.

(h) Very carefully lower decant leg into crystallizer until decant leg reaches crystal bed then decant crystallizer solution to designated tank. (C-513 decants to C-613. C-613 jets to C-17 if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613). If any crystals are decanted from C-513 to C-613, transfer back to C-513.

(i) Turn off the chilled water to crystallizer coil.

(j) Crystallizer is ready for next batch of feed solution or filtrate from primary crystallizer.

NOTE: Control all crystal beds to between 4 and 10 liters.

(k) Continue transferring from C-38 and doing crystallization procedures until C-38 is empty.

(l) Reduce the product crystal bed in C-513 with water crystallizations until there is approximately 4 liters. Check crystal volume with sampler. Decant each H_2O crystallization to C-613.

(m) Jet C-613 to C-17. (If there is a crystal bed in C-613, add enough water to dissolve all crystals before jetting to C-17.)

5. Rubidium Removal Procedure

(a) First Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(b) Second Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(c) Third Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

   NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(d) Fourth Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

   NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(e) Fifth Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

   NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(f) Sixth Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

   NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available, and if C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(g) Seventh Alum Addition

1. Add water to C-513 to a volume of 25 liters.

2. Add 300 grams of ammonium alum to C-513.

3. Heat C-513, with agitation, to 90°C for 15 minutes.

4. Cool to 25°C.

5. Turn off agitator and settle for 15 minutes.

   NOTE: The transfer pump must always be primed with water before starting pump.

6. Decant supernate to C-613 via transfer pump.

7. Jet C-613 to C-17, if C-17 is available. If C-17 is not available, make scavenge crystallization in C-613. When C-17 becomes available, dissolve crystal bed and transfer to C-17.

(h) After the last decant from C-513 on the seventh addition, filter the remainder of the supernate via FV-2 to C-613.

(i) Add 10 liters of cold water to C-513. Turn on chilled water to coil and agitate solution for two minutes. Settle the crystals.

(j) Filter the supernate via FV-2 to C-613. Suck crystal bed dry with the filter.

6. Cesium-137 Product Crystal Dissolution

(a) Drain the Cell 11 hot off gas scrubber.

(b) Check volume of 1.5 M NaOH in off gas scrubber head tank and adjust flow rate to scrubber to 10 liters/hr, 85% setting on rotometer.

(c) With off gas on the evaporator, add 15 liters of 0.5 N HCl to C-513. Follow with a one liter H_2O wash. (Make-up = 625 ml of 12 N HCl diluted to 15 liters with water.) Use the addition funnel at make-up Area 33.

7. Precipitation of Cesium With Platinum

NOTE: If any precipitate goes to transfer vessels during a transfer, it must be returned to C-513.

(a) Heat the crystallizer with agitation to 90°C.

(b) Add slowly to the crystallizer over a five minute period, 500 grams of platinum as H_2PtCl_6.

(c) Continue digesting with agitation at 90°C for an additional 30 minutes.

(d) Cool with agitation to 20°C.

(e) Turn off agitation and allow the solution to settle for 30 minutes or until all precipitate has settled out.

NOTE: Check that C-613 is empty and rinsed of all alum crystals to C-17.

(f) Transfer solution, via filter and FV-2, to C-613. (To be processed later for recovery of the platinum.)

(g) First HCl Acid Rinse

1. Add 10 liters of cool 6 N HCl to C-513. (Five liters of 12.0 N HCl built up to 10 liters with water.) Add via additional funnel at Area 33.

2. Agitate C-513 solution for 5 minutes. Then allow the precipitate to settle.

3. Filter the solution from C-513 to FV-2 transfer vessel.

4. NOTE: E-213 maximum operating capacity is 12.0 liters. DO NOT EXCEED. Do not exceed 8 psi steam pressure to E-213.

5. Drain FV-2 to E-213 and boil down by a factor of 2 to 1. Make sure that E-213 off gas never goes below 3 inches and water is on the E-213 off gas condenser during evaporation.

(h) Second HCl Acid Rinse

1. Add 10 liters of cool 6 N HCl to C-513. (Five liters of 12.0 N HCl built up to 10 liters with water.) Add via additional funnel at Area 33.

2. Agitate C-513 solution for 5 minutes. Then allow the precipitate to settle.

3. Filter the solution from C-513 to FV-2 transfer vessel.

4. NOTE: E-213 maximum operating capacity is 12.0 liters. DO NOT EXCEED. Do not exceed 8 psi steam pressure to E-213.

5. Drain FV-2 to E-213 and boil down by a factor of 2 to 1. Make sure that E-213 off gas never goes below 3 inches, and water is on the E-213 off gas condenser during evaporation. If E-213 is full, drain FV-2 to C-613.

(i) Third HCl Acid Rinse

1. Add 10 liters of cool 6 N HCl to C-513. (Five liters of 12.0 N HCl built up to 10 liters with water.) Add via additional funnel at Area 33.

2. Agitate C-513 solution for 5 minutes. Then allow the precipitate to settle.

3. Filter the solution from C-513 to FV-2 transfer vessel.

4. NOTE: E-213 maximum operating capacity is 12.0 liters. DO NOT EXCEED. Do not exceed 8 psi steam pressure to E-213.

5. Drain FV-2 to E-213 and boil down by a factor of 2 to 1. Make sure that E-213 off gas never goes below 3 inches and water is on the E-213 off gas condenser during evaporation. If E-213 is full, drain to C-613.

(j) Boil down HCl rinses to 6 liters and transfer to C-613.

(k) Add 5 liters of cool water to C-513 (do not agitate). Rinse walls of C-513 while adding water.

NOTE: The 5 liters H_2O rinse from C-513 should follow the HCl boil down in E-213 and C-613.

(l) Filter C-513 solution via FV-2 transfer vessel to E-213 when evaporator is empty. Then transfer to C-613.

(m) Rinse E-213 with two 1-liter portions of H_2O to C-613. Do not exceed 35 liters of C-613.

(n) After transferring E-213 to C-613, agitate and sample, _____ C13W.

NOTE: Mark sample sheet and bottle that this solution contains HCl. Hold for Analysis. Hold this solution in C-613 for Cs^{137} and platinum recovery.

8. Cesium Chloroplatinate Dissolving

(a) Add 15 liters H_2O to C-513.

(b) Turn on C-513 agitator and chilled water to coils.

(c) Cool C-513 solution to 20°C. Leave chilled water on coils.

(d) Add 2/3rds calculated amount of 42.5% hydrazine hydrate very slowly while controlling the temperature below 30°C.

NOTE: The amount of hydrazine hydrate to add 1.5 liters of 42.5% hydrazine hydrate. (3 ml for each gram of platinum added in the Precipitation Procedure).

(e) Turn off water to coils.

(f) Digest the solution with agitation at 25°C for 30 minutes.

(g) Add 1/3 of calculated amount of 42.5% hydrazine.

(h) Heat slowly over a period of 15 minutes to 95°C. Then digest at 95°C for two hours while maintaining volume at approximately 16 liters by adding water. Watch C-513 off-gas line for condensate plug.

(i) Cool solution to 25°C, turn off agitator and allow all precipitate to settle.

9. Cesium-137 Product Evaporation

(a) Check that Cell 13 hot off gas head vessel has 1.5 M NaOH and flow meter rate is 85% setting on the rotometer.

(b) Turn on water to evaporator hot off gas condenser and check that off gas to evaporator is 5 to 8 inches. Also check that E-213 is empty and rinsed.

(c) Filter product solution from the crystallizer via FV-2 vessel. Make test for platinum precipitate.

NOTE: Drain any black filtrate back to C-513 and allow to settle loner.

(c) Cont'd

Platinum Test

1. Use 10 ml of filtered supernate.
2. Add 1 ml of hydrazine.
3. If precipitate forms, it is a platinum compound and will need more hydrazine in C-513.
4. Add 100 ml increments, digest and repeat test.

(d) If no platinum is found in supernate, filter 8.0 liters to FV-2 and drain to E-213.

(e) Add 800 ml of concentrated HCl to E-213.

(f) Add 1.2 liters of 70% HNO_3 to E-213.

(g) Close the evaporator by inserting plug.

(h) Turn on the steam to evaporator jacket. Do not exceed 8 psi on E-213 steam header. Heat to 80°C, cut off steam until temperature levels out. Heat to 95°C and cut off steam until temperature levels out. Heat to 100°C. Watch vapor line for burping solution. If everything is okay, continue with Step (i).

(i) Evaporate to 2.0 liter volume. Then cool the evaporator to 30°C.

(j) Repeat Steps (d) through (h).

(k) Evaporate E-213 solution to 4.0 liters and then cool E-213 to 30°C.

(l) Rinse C-513 with 4 liters of 1 N HCl and transfer to evaporator. Reduce volume to 4 liters.

(m) Repeat Step (l) and reduce volume to 3 liters.

(n) Cool evaporator to 60°C and add 3 liters of concentrated HCl to the evaporator.

(o) Evaporate to 3 liter volume.

10. Destruction of Ammonium Salts with Aqua Regia

(a) Cool evaporator to 30°C for first HNO_3 addition.

NOTE: Each addition of HNO_3 should be added slowly.

(b) Add 100 ml of 70% HNO_3 to evaporator.

(c) Very slowly add 4 liters of concentrated HCl to evaporator.

(d) Digest at 45°C for 30 minutes.

(e) Very carefully heat to 100°C and boil evaporator solution for 15 minutes.

(f) Cool evaporator to 75°C and add 100 milliliters of 70% HNO_3 to evaporator.

(g) Boil evaporator solution for 15 minutes.

(h) Cool evaporator to 75°C and add 100 milliliters of 70% HNO_3 to evaporator.

(i) Boil evaporator solution for 15 minutes.

(j) Cool evaporator to 75°C and add 100 milliliters of 70% HNO_3 to evaporator.

(k) Boil evaporator for 15 minutes.

(l) Cool evaporator to 75°C and add 100 milliliters of 70% HNO_3 to evaporator.

(m) Heat evaporator and reduce the volume to 3 liters.

(n) Cool evaporator to 30°C.

11. Removal of HNO_3 and HCl From the Cesium Product

(a) Very slowly add 2 liters of concentrated HCl to evaporator.

(b) Reduce evaporator volume to 3 liters. Assume specific gravity of _____.

(c) Cool evaporator to 60°C and add 2 liters of concentrated HCl to the evaporator.

(d) Reduce evaporator volume to 2 liters. Then cool to 60°C.

Assume Specific Gravity of _____

(e) Add 2 liters of distilled water to the evaporator.

(f) Reduce evaporator volume to 1.5 liters. Then cool to 60°C.

Assume Specific Gravity of _____

(g) Add 2 liters of distilled water to the evaporator.

(h) Reduce evaporator volume to 1.5 liters and then cool to 60°C.

(i) Add 2 liters of distilled water to the evaporator.

(j) Reduce evaporator volume of 2 liters. Then cool to 60°C for Procedure No. 12.

Assume Specific Gravity of _____

12. Cesium-137 Powdering Operation

(a) Clean off drying tray thoroughly and plug drain in case of breakage.

(b) Check that the FV-2 transfer vessel is clean and dry.

NOTE: Split final volume into 2 batches, since 10,000 curies of powder is all that can be handled in drying beaker.

(c) Check that a clean filter is in line from the evaporator to FV-2.

(d) Filter 500 milliliters of Cs^{137} product to FV-2. If more than 500 milliliters is transferred to FV-2, return excess to evaporator to prevent drain line from plugging.

(e) Drain 500 milliliters of product from FV-2 to Vycor beaker. Do not exceed 500 milliliters in drying beaker at one time.

(f) Place off-gas funnel over Vycor beaker and outlet line into E-213 plug hole.

(g) Heat Vycor beaker at a variac setting of 70 on No. 2 circuit.

(h) Boil off solution very slowly and carefully (check hot plate for heating by a drop of water on it).

(i) When solution gets syrupy, turn off heat and stir constantly and carefully with tantalum stirrer. If the solution is not stirred carefully, it will cake and the whole process will have to be repeated.

NOTE: After powder is dry, transfer to hot plate and bake 300 to 400°C for four hours. Stir occasionally to prevent caking.

(j) Transfer powder to Cell 14 and sample _____ CsP with measuring spoon.

NOTE: Mark sample sheet and bottle that this solution contains HCl.

(k) Weigh powder and transfer to the stainless steel storage cans.

NOTE: Use wrench to tighten can cap snugly.

(l) Fill out powder data card and place card in hole rack that corresponds with well number that can has been placed in.

NOTE: When viewing storage well through mirror Row "B" will be in top half of mirror, Row "A" will be in bottom half of mirror.

13. Platinum Recovery and Conversion to H_2PtCl_6

CHECK WITH SUPERVISION FOR STEP (a) or (j)

(a) Boil down platinum recovery solution to 3.0 liters in E-213.

(b) Transfer the solution from C-613 to C-513.

(c) Turn on C-513 agitator and water to coil. Titrate to less than 0.1 N HCl.

(d) Slowly add calculated amount of hydrazine hydrate to C-5 while controlling temperature below 50°C. The calculated amount of hydrazine equals one-half the amount of the hydrazine that was added during the process.

(e) Digest solution at 25°C for 30 minutes.

(f) Heat slowly over a period of 15 minutes to 90°C. Digest at 90°C for 45 minutes.

(g) Cool solution to 25°C. Turn off agitator and settle precipitate.

NOTE: Check with supervision for Step (h).

(h) Transfer supernate to HCl drain via C-613 filter and FV-2 transfer vessel. Make test for platinum in the supernate.

(i) Rinse C-513 with 15 liters of 0.5 N HCl. Agitate 5 minutes, let settle for 15 minutes, and then filter to HCl Boltaron drain via FV-2 transfer vessel.

(j) Repeat Step (i) two more times.

(k) Add 10 liters of concentrated HCl, via Area 33 funnel to C-513. Then rinse and turn on agitator.

(l) Add 2.0 liters of 30% H_2O_2 via wall funnel to C-513 in 100 milliliter increments. Wait 10 minutes between additions.

(m) Digest C-5 solution until all platinum is in solution. To determine if platinum is all in solution, transfer to FV-2 transfer vessel and check visually. More HCl and H_2O_2 may be added if platinum is not in solution. For every liter of additional HCl added, add 200 milliliters of H_2O_2 in 100 milliliter increments.

NOTE: Do not exceed 12 liters total in evaporator.

(n) Transfer C-513 solution to the evaporator via FV-2.

(o) Add one liter of water to C-5 and transfer to evaporator.

NOTE: Do not exceed 8 psi of steam on evaporator.

(p) Evaporate volume down to two liters.

(q) Then cool to 60°C.

(r) Transfer the evaporator solution to FV-2 transfer vessel and then to platinum bottles.

(s) Sample the solution for the Lab. Code_____Ptl. This sample goes to Musick's lab.

NOTE: Mark sample sheet and bottle that this solution contains HCl.

(t) Sample S-424 for Cs^{137} and platinum. Code_____Cl4W. Check pH and add NaOH if solution is acid.

NOTE: Mark sample sheet and bottle that this solution contains HCl.

APPENDIX NO. 5-q

F3P CHEMICAL MAKE-UP - BUILDING 3517 PROCEDURE

I. Protective Equipment

(a) All operating personnel will wear the following protective equipment during chemical make-up.

1. Face shields.
2. Rubber gloves.
3. Coveralls with sleeves fastened at the wrist.
4. Safety shoes.
5. Respirators while handling dry chemicals.

II. Operating Procedure

(a) Premake-up checks:

1. Empty and flush make-up tank with water.
2. Add required quantity of water to the tank and cool to a temperature of 20°C.
3. Cooling water on the tank coils.
4. Mechanical agitator operating and giving the proper agitation to the solution.
5. Tank ventilation system is on and all other tank solid addition lids are closed. Check for a definite air sweep into the make-up tank being used.
6. Turn off water addition valve to prevent overflowing tank while adding chemicals.

(b) Make-up:

1. Add all dry chemicals with a scoop or shovel and hold tank temperature below 50°C.
2. 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.

(c) Chemical addition to process cell tanks:

1. All chemical addition to process cell tanks will be followed by a water flush through the addition funnel. The quantity of water added will be determined by supervision.

(d) Hydrazine:

1. Hydrazine will be dispensed under the supervision of the building foremen.

2. Hydrazine will be stored and dispensed in designated areas. These areas are located at a definite distance from nitric acid storage or make-up.

3. Hydrazine drums are to be placed on the drum holders and the solution drained into the measuring container from this device. No other method is acceptable.

4. The measuring container is a calibrated six-liter stainless steel bucket which is to be used for hydrazine only.

5. During the transfer of hydrazine from the carboy to the process vessel solution addition funnel, the area will be chained off with "No Smoking" signs and smoking is prohibited in this area.

6. The hydrazine carboys will be valved and locked closed when not in use.

7. Dilution of hydrazine solution will be done in the chemical fume hood.

(e) Potassium permanganate ($KMnO_4$):

1. $KMnO_4$ solutions will be made up in M-3 tank only and will be used only as required in Cell 9 solvent extraction operations.

2. $KMnO_4$-NaOH solution for decontamination will be made up as follows:

Add water to make-up tank.

Add $KMnO_4$ to make-up tank.

Add NaOH to make-up tank.

(f) Solvents:

1. Smoking is prohibited while handling solvents and all areas where solvent is used will be chained and tagged.

2. Solvents will be stored in the F3P solvent storage shed.

3. All dispensing drums will be grounded by clamping the ground wire to the drum.

4. When draining solvent into a safety can, the safety can will be grounded to the dispensing drum.

5. All solvents will be transported to the building in approved safety cans.

6. All solvent addition to process vessels will be done from the safety cans and the ground wire from the can will be clamped to the funnel addition system.

APPENDIX NO. 5-r

SHIELDED TRANSFER TANK HANDLING PROCEDURE

1. Remove cupola cover.

2. Survey cupola.

3. Remove covers from long leg, vent line and fill line.

4. Survey exposed couplings. Wrap wipes around the lines to prevent contamination when disconnecting.

5. Connect hot off-gas line to the vent line and record the vacuum or pressure in the tank.

6. Vent the tank to the hot off gas system.

7. Connect the proper transfer line to the long leg.

8. Rotate the movable section of the long leg to the right, ~ 45°. (Use special wrench to rotate.) Push down and rotate 45° to the right. This locks the long leg in position for emptying.

9. Connect the water line to the fill line on the tank.

10. Check all fittings to make sure the connections are secure.

11. Open vent valve and close hot off gas valve.

12. Open valve on transfer line and vacate the cell. Transfer of solution is controlled from the panelboard.

13. After the transfer is complete (temperature rise on jet or air bubbles in tygon transfer line) rinse tank by adding four 10-second water rinses.

14. Enter cell under the regulations for Personnel Entry into F3P Cells.

15. Rotate long leg to the left, ~ 45°. Raise to upper position and rotate to the left, ~ 45°. This locks the long leg in the vent position. Disconnect lines carefully to prevent solution from dripping into cupola or on the outside of the tank.

16. Install a rubber stopper and wire down on the end of the long leg. (The Snaptite valve will not hold under vacuum applied at the fill site.)

17. An access nozzle to the area between the inner tank and the outer tank is located in the cupola. It is recommended that each tank be tested between shipments. Evacuate the S.T.T. to 28" Hg vacuum and hold for 30 minutes. If a leak is noted from loss of vacuum on the gauge, a check should be made to determine the exact location of the leak.

17. (Cont'd)

NOTE: The Snaptite valves will not hold under vacuum and should be capped off with a rubber stopper.

18. Replace caps on long leg, vent line and fill line.

19. Survey cupola and decontaminate if necessary.

20. Install cover on cupola and be sure gasket is in good condition.

21. Survey and smear container before removing it from the building.

APPENDIX No. 5-s

PROMETHIUM PURIFICATION CHEMICAL PROCESS DESCRIPTION

The quantity of Pm^{147} normally processed in the Cell 10 equipment during a run will be 75,000 curies.

Rare Earths Separation (Nd-Pm Split)

The separation of the rare earths, Nd-Pm-Sm, depends on the difference in their extractibility by 100% TBP from strong nitric acid solution. The higher the atomic number the greater their extractibility. Higher nitric acid concentrations increase the magnitude as well as the difference in their extractibility.

In the Nd-Pm split (see Appendix No. 4-a for the flowsheet) the aqueous phase entering the 20-stage cascade is 9 M HNO_3 (16.46 l/hr) and the organic phase entering at the opposite end of the cascade is 100% TBP (23 l/hr). The feed enters the cascade at stage 8 as rare earth nitrates (25 g/l in 14 M HNO_3) at a rate of 1 l/hr. The 20 M HNO_3 reflux acid (7 l/hr) enters the cascade at stage 13. The aqueous phase leaving the cascade contains 99.88% of the Nd and 4.6% of the Pm. The organic phase leaving the cascade contains 95.1% of the Pm, 0.12% of the Nd and essentially all of the Sm and Eu.

The Pm-Sm-Eu-containing organic stream leaving the 20-stage cascade passes to an 11-stage stripper cascade where the rare earths are removed from the organic stream by continuous counter-current contacting with water (12 l/hr).

The aqueous stream from the 11-stage stripper is continuously evaporated and at the end of the run is made to 14 M in HNO_3 and 25 g/l rare earth for feed in the Pm-Sm split.

Rare Earths Separation (Pm-Sm Split)

In the Pm-Sm cycle (see Appendix No. 4-a for the flowsheet) the feed (1 l/hr) and the 20 M HNO_3 reflux acid (7.0 l/hr) enter the cascade at stage eight. The other streams are the same as the Nd-Pm cycles except the aqueous stream flow rate is 17.4 l/hr.

Ninety-five percent of Pm and 0.2% of the Sm leave the cascade in the aqueous stream (raffinate).

The raffinate is washed to remove TBP and continuously evaporated to obtain the final product.

The organic stream is stripped of rare earths in the 11-stage mixer-settler and the organic is recycled.

PROMETHIUM PURIFICATION OPERATING PROCEDURE

NOTE: If the last separation done in the cascade was of materials other than rare earths, it is necessary to flush and decontaminate all equipment used in the previous run. If the last separation done in the cascade was of rare earths, proceed beginning with Section "B."

I. Promethium-Neodymium Separation

A. Cascade Clean Up

(1) Solution make up tanks.

(a) Empty tanks M-833, M-1333, R-510, R-210, R-410 and calibration burettes.

(b) Rinse tanks M-833, M-1333, R-510, M-833 calibration burettes and feed tank air lifts.

(c) Fill M-833 and M-1333 with distilled water.

(2) Drain cascade raffinate to R-110 and stripper aqueous to R-110 through E-210 by lowering weirs on X-110 and stripper to minimum level.

(3) Turn on agitators to X-210, X-110, W-210 and stripper and adjust to maximum speed.

(4) Wash the acid from the cascades and stripper by maximum flow of H_2O from P-13 and 15 liters/hr from tank M-1333.

(5) Continue to wash until samples taken from weir boxes are above pH 5.

(6) Wash out W-110 and E-110 by diverting raffinate stream from R-110 to E-110. Wash for 30 minutes. Change X-110 raffinate stream to R-110 and jet E-110 to E-210.

(7) Shut off flow of H_2O from M-833 and M-1333 and drain.

(8) Fill tanks M-833 and M-1333 with 0.2 M NaOh and fab detergent solution.

(9) Wash organic from X-110, X-210 and stripper with maximum flow from P-13 and 15 liters/hr from M-1333. The flow from X-110 goes directly to R-110, and the flow from the stripper goes through E-210 to R-110.

(10) After 3-hours operation pull sample from X-110 weir, add 2 ml of HNO_3 to sample and check for organic phase. If organic is visible, sample each hour until no organic is visible in sample.

(11) Shut off flow from M-833 and M-1333. Drain and rinse M-833 and M-1333 and fill with distilled H_2O.

(12) Wash the emulsion from X-210, X-110 and stripper with maximum flow from P-13 and 15 liters/hr from M-1333.

(13) Continue wash until X-110 raffinate and strip aqueous are less than pH 11.

(14) Close down cascade and drain all solution make up tanks.

B. Solution Make Up

(1) Reflux acid solution. Make up ~100 liters of 20.00 ± 0.05 M HNO_3 in tank M-1233.

NOTE: Only one day's supply of 20.00 M HNO_3 should be made up at one time. Check and adjust normality on 20.00 M HNO_3 that has been made up more than 24 hours.

(2) Strip aqueous solution. Fill tank M-1333 with distilled H_2O.

(3) Cascade aqueous solution. Make up 9.00 ± 0.02 M HNO_3 in M-833.

(4) Raffinate wash liquid. Add Amsco as required to M-933.

(5) Cascade organic liquid. Add 100% TBP to tank R-410.

(6) Rare earth feed solution.

(a) Transfer rare earth feed solution from Cell 9 to R-510.

(b) Sample R-510, determine rare earth concentration and normality.

(c) Adjust feed solution in R-510 to 14.0 ± 0.5 M HNO_3 and 25 grams rare earth (as metal)/liter.

C. Aqueous Organic Equilibration

(1) Valve X-110 raffinate to R-110 and stripper aqueous to E-210 to R-110.

(2) Set X-110 and stripper weirs to maintain normal interface.

(3) Start P-13 and adjust for maximum flow.

(4) Start P-11 and adjust for maximum flow. Pump 100% TBP from R-410. Refill R-410 as required.

(5) Adjust flow of aqueous from M-1330 to stripper to 12 liters/hour.

(6) Adjust Amsco flow to W-210 and W-110 to 2 liters/hour.

(7) Start agitators to X-110, X-210, stripper, W-110 and W-210.

(8) Continue flow of cascade aqueous, organic and stripper aqueous until organic begins to leave stage 20 of X-210. (Indicated by probes).

(9) Reset P-11 to deliver 23 liters/hour.

(10) Reset P-13 to deliver 16.46 liters/hour.

(11) Reset stripper aqueous valve to deliver 12 liters/hour.

(12) Valve tank M-233 to stage (13) of X-210. Set P-1 to deliver 7 liters/hour and start P-1 pump.

(13) Collect TBP in R-210 for (3) hours. Drain R-210 to disposal pump. Add 80 liters of TBP to R-210. Change TBP flow to P-11 from (R-410 to R-210).

(14) Check density of raffinate at weir box. If the cascade is in acid equilibrium specific gravity will be 1.300 grams/ml.

(15) If the specific gravity of the raffinate is less than 1.300 grams/ml continue operating. Check pumps, flows and sample raffinate each hour until a specific gravity of 1.300 grams/ml is obtained.

(16) Empty R-110 and close gravity drain valve from E-210 to R-110.

D. Nd-Pm Fractionation

(1) Valve T-210 to stage (8) and R-510 to primary air lift.

(2) Open air supply to primary air lift and check flow with Metering Burette.

(3) Open air supply to secondary air lift (low flow) and set to maintain flow of feed to stage (8) at 1,000 ml/hour.

(4) Turn on condenser H_2O to E-210 and check off-gas.

(5) Turn on steam to E-210 jacket and set E-210 temperature controller at 120°C.

(6) One hour checks and adjustment to be made during operation.

(a) P-1, P-11 and P-13 flows.

(b) Interface levels in cascade and stripper.

(c) Motor or bearing noise.

(d) Primary and secondary air lift deliveries.

(e) E-210 temperature and off-gas.

(7) Continue operating until R-510 is empty.

E. Feed Tank Rinse and Pm-Nd Wash Out Cycle

(1) Add four (4) liters of 14 M HNO_3 to R-510. Sparge for 5 minutes and feed to stage 8 at the rate of 1,000 ml/hour.

(2) When R-510 is empty, sample the aqueous from the stripper and determine promethium concentration.

(3) While waiting for results, repeat steps 1 and 2 as required until allowable results are obtained.

F. Equipment Shut Down

(1) Turn off P-1, P-11 and P-13 pumps.

(2) Turn off air to feed air lifts.

(3) Turn off X-110, X-210, W-210 and stripper agitators.

(4) Turn off steam to E-210 jacket and H_2O to E-210 condenser.

(5) Close all valves.

(6) Sample E-210 and R-110. If R-110 is under allowable concentration, jet to waste header.

II. Promethium-Samarium Separation

A. Solution Make Up

(1) Reflux acid solution. Make up ~100 liters of 20.00 ± 0.05 M HNO_3 in tank M-1233.

(2) Strip aqueous solution. Fill tank M-1333 with distilled H_2O.

(3) Cascade aqueous solution, make up 9.00 ± 0.02 M HNO_3 in M-833.

(4) Raffinate wash liquid. Add Amsco as required to M-933.

(5) Cascade organic liquid. Add 100% TBP to R-210 if necessary.

(6) Add 1/3 calculated amount of 14 M HNO_3 required to dilute concentrate to 25 grams/liter as metal to E-210 and jet to R-510.

(7) Use the remaining 2/3 calculated 14 M HNO_3 as two separate washes for E-210. Jet each wash to R-510.

(8) Adjust specific gravity of solution in R-510 to 1.385 by addition of fuming HNO_3 or H_2O as required.

B. Cascade Start Up

(1) Set pump P-11 to deliver 23.0 liters/hour. Open proper valves and start pump.

(2) Set pump P-13 to deliver 17.4 liters/hour. Open proper valves and start pump. Flow raffinate solution to R-110.

(3) Set P-1 pump to deliver 7 liters/hour. Open proper valve to tank and stage (8) and start pump.

(4) Set weirs for normal operation.

(5) Open valves and adjust flow from M-1333 to stripper at 12 liters/hour. Solution in E-210 draining to R-110.

(6) Open valves and adjust flow M-933 to W-110 and W-210 at 2 liters/hour.

(7) Start agitators to W-110, W-210, stripper, X-110 and X-210.

(8) Continue operation for four (4) hours.

(9) Close E-210 drain to R-110 and jet R-110 to waste header.

C. Promethium-Samarium Fractionation

(1) Change raffinate flow from R-110 to E-110.

(2) Turn on condenser H_2O to E-110 and E-210 condensers and check off-gas.

(3) Turn on steam to E-110 and E-210 and set temperature controllers at 120°C.

(4) Valve T-210 to stage (8) and R-510 to primary air lift.

(5) Open air supply to primary air lift and check flow with Metering Burette.

(6) Open air supply to secondary air lift (low flow) and set to maintain flow of feed to stage (8) at 1,000 ml/hour.

(7) One hour checks and adjustments to be made during operation.

(a) P-1, P-11 and P-13 flows.

(b) Interface levels in cascade and stripper.

(c) Motor or bearing noise.

(d) Primary and secondary air lift deliveries.

(e) E-110, E-210 temperature and off-gas.

(8) Continue operation until R-510 is empty.

D. Wash Out Cycle After Pm-Sm Fractionation

(1) Add 4 liters of 14.0 M HNO_3 to R-510. Sparge for 5 minutes and feed to stage 8 at the rate of 1,000 ml/hour.

(2) When R-510 is empty, sample raffinate for G.B. to determine allowable promethium concentration.

(3) While waiting for results repeat steps 1 and 2 as required until allowable results are obtained.

E. Equipment Shut Down

(1) Turn off P-1, P-11 and P-13 pumps.

(2) Turn off air to feed air lifts.

(3) Turn off X-110, X-210, W-110, W-210 and stripper agitators.

(4) Turn off steam to E-110, E-210 and condenser H_2O to E-110 and E-210.

(5) Close all valves.

(6) Sample E-110 and E-210.

APPENDIX No. 5-t

STRONTIUM-CALCIUM SEPARATION CHEMICAL PROCESS DESCRIPTION

The quantity of Sr^{90} normally processed in Cell 10 during a run will be 20,000 curies.

The separation of Ca from Sr (see Appendix No. 4-b for the flowsheet) depends on the preferential extraction of Ca into a 0.4 M D2EHPA - Amsco phase. An additive, 2-ethyl hexanol, is used in the organic phase to reduce the change in the distribution coefficients due to varying degrees of loading. The 2-ethyl hexanol also reduces the distribution coefficients, and thus allows the use of a higher concentration of D2EHPA without excessively high distribution coefficients. The hydrogen ion concentration is maintained in operable limits by use of an acetate system.

The aqueous phase is 0.1 M HAC (25 l/hr). The organic phase (25 l/hr) is 0.4 M D2EHPA and 2.5 M 2-ethyl hexanol diluted with Amsco. The feed (3.3 l/hr) contains 30 g/l Ca and 1.5 g/l Sr as acetates.

The strontium product is recovered by continuous evaporation of the raffinate. The calcium and yttrium are stripped from the organic phase and the organic is recycled.

Sr-Ca SEPARATION OPERATING PROCEDURE

NOTE: If the last separation done in the cascade was of materials other than Sr^{90}, it is necessary to flush and decontaminate all equipment used in the previous run. If the last separation done in the cascade was Sr^{90}, proceed beginning with Section "B."

A. Cascade Clean Up

(1) Solution make up tanks.

(a) Empty tanks M-833, M-1333, R-510, R-410, R-210 and calibration burettes.

(b) Rinse tanks M-833, M-1333, R-510, R-410, R-210, calibration burettes and feed tank air lifts.

(c) Fill M-833 and M-1333 with distilled H_2O.

(2) Drain cascade raffinate to R-110 and stripper aqueous to R-110 through E-210 by lowering weirs on X-110 and stripper to minimum level.

(3) Turn on agitators to X-210, X-110 and W-210 and stripper and adjust to maximum speed.

(4) Wash the acid from the cascades and stripper by maximum flow of H_2O from P-13 and 15 liters/hour from tank M-1333.

(5) Continue to wash until samples taken from weir boxes are above pH 5.

(6) Wash out W-110 and E-110 by diverting raffinate stream from R-110 to E-110. Wash for 30 minutes. Change X-110 raffinate stream to R-110 and jet E-110 to E-210.

(7) Shut off flow of H_2O from M-833 and M-1333 and drain.

(8) Fill tanks M-833 and M-1333 with a 0.2 M NaOH and fab detergent solution.

(9) Wash organic from X-110, X-210 and stripper with maximum flow from P-13 and 15 liters an hour from M-1333. The flow from X-110 goes directly to R-110 and the flow from the stripper goes through E-210 to R-110.

(10) After three hours operation pull sample from X-110 weir, add 2 ml of HNO_3 to sample and check for organic phase. If organic is visible, continue operating and sample each hour until no organic is visible.

(11) Shut off flow from M-833 and M-1333. Drain and rinse M-833 and M-1333 and fill with distilled H_2O.

(12) Wash the emulsion from X-210, X-110 and stripper with maximum flow from P-13 and 15 liters/hour from M-1333.

(13) Continue wash until X-110 raffinate and stripper aqueous solution are less than pH 10.

(14) Close down cascade and drain all solution make up tanks.

B. Solution Make Up

(1) Cascade aqueous solution. Make up 0.1 M acetic acid in M-833.

(2) Stripper aqueous solution. Fill tank M-1333 with 1 M HNO_3.

(3) Raffinate wash liquid. Add Amsco as required to M-933.

(4) Cascade organic liquid. Fill M-410 with 0.4 M D2EHPA, 2.5 M 2-eth. hex and Amsco.

(5) Strontium feed solution. Transfer from strontium cell to R-510 and adjust feed solution to 1.5 grams Sr/liter and 30 grams Ca/liter. The acetic acid should not be more than 25% of the total acetate present.

C. Aqueous-Organic Equilibration

(1) Valve X-110 raffinate to R-110 and stripper aqueous to E-210 to R-110.

(2) Set X-110 and stripper weirs to maintain normal operating interface.

(3) Start P-13 and adjust for maximum flow.

(4) Start P-11 and adjust for maximum flow. Pump D2EHPA from R-410. Refill R-410 as required.

(5) Adjust flow of aqueous from M-1330 to stripper to 12 liters/hour.

(6) Adjust Amsco flow to W-110 to 2 liters/hour.

(7) Start agitators to X-110, X-210, stripper and W-110 at normal operating speed.

(8) Continue flow of cascade aqueous, organic and stripper aqueous until organic begins to leave stage 20 of X-210. (Indicated by probes).

(9) Reset P-11 to deliver 25 liters/hour.

(10) Reset P-13 to deliver 25 liters/hour.

(11) Reset stripper aqueous valve to deliver 12 liters/hour.

(12) Collect D2EHPA in R-210 for three hours. Drain R-210 to disposal pump. Add 80 liters of 0.4 M D2EHPA, 2.5 M 2-eth. hex. and Amsco to R-210. Change flow to P-11 from (R-410 to R-210).

(13) Check pH of raffinate. Should be between 3.5 and 1. If the pH is greater than 3.5, continue operating and sample raffinate each hour until it is within limits.

(14) Empty R-110 and divert raffinate stream from R-110 to E-110.

D. Calcium-Strontium Fractionation

(1) Valve T-210 to stage 9 and R-510 to primary air lift.

(2) Open air supply to primary air lift and check flow with metering burette.

(3) Open air supply to secondary air lift (high flow) and to maintain flow of feed to stage 9 at 3.33 liters every hour.

(4) Turn on condenser H_2O to E-110 and check E-110 off-gas.

(5) Turn on E-110 steam to E-210 jacket and set E-110 temperature controller at 120°C.

(6) One hour checks and adjustments to be made during operation.

(a) P-11 and P-13 flows.

(b) Interface levels in cascade and stripper.

(c) Motor or bearing noise.

(d) Primary and secondary air lift deliveries.

(e) E-110 temperature and off-gas.

(7) Continue operating until R-510 is empty.

E. Feed Tank Rinse and Calcium-Strontium Wash Out Cycle

(1) Add 7 liters of 0.1 M acetic acid to R-510. Sparge for 5 minutes and feed to cascade at the rate of 3.3 liters/hour.

(2) When R-510 is empty, sample raffinate for G.B. to determine allowable strontium concentration.

(3) While waiting for results, repeat steps 1 and 2 as required until allowable results are obtained.

F. Equipment Shut Down

(1) Turn off P-11 and P-13 pumps.

(2) Turn off air to feed air lifts.

(3) Turn off X-110, X-210, W-110 and stripper agitators.

(4) Turn off steam to E-110 jacket and H_2O to E-110 condenser.

(5) Close all valves.

(6) Sample E-110 and R-110. If R-110 is under allowable concentration, jet to waste header.

APPENDIX NO. 6

ZONING PROCEDURE FOR THE FISSION PRODUCT PILOT PLANT

I. Regulated Zone

Definition: 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.

A. The F3P area is a regulated zone and signs to this effect are posted at all entrances to the area.

2. Contamination Zone

Definition: An area where control measures involve the contamination of employees, the environs, and/or equipment and where there is a possibility that radioactive material may become deposited inside the body leading to internal radiation exposure. See Table 1 for action required for specific instances.

A. F3P Area Contamination Zones

(a) Process cells.

(b) Manipulator cells.

(c) Tank Farm cells.

(d) Cells 15, 16, 17, 18 and 19.

(e) Service and pipe tunnels.

(f) Vacuum pump cubicle.

(g) Filter pit.

(h) Tank Farm sampler.

(i) Hot can storage shed.

(j) Cubicle "D".

(k) Other contamination zones may be designated on a temporary basis as needed.

Table 1 - Contamination Levels to be Used as a Guide in the Establishment of Contamination Zones

Type of Radiation	Airborne Contamination	Direct Reading Surface Contamination	Transferable Surface Contamination
Alpha	5×10^{-11} uc/cc air	300 d/m/100 cm^2	30 d/m/100 cm^2
Beta-Gamma	1×10^{-8} uc/cc air	0.25 mrad/hr	1000 c/m/100 cm^2

3. Radiation Zone

Definition: An area where control measures involve external radiation exposure to personnel. See Table 2 for action required in specific instances.

A. F3P Area Radiation Zones

(a) Process cells.

(b) Manipulator cells.

(c) Tank Farm cells.

(d) Cells 15, 16, 17, 18 and 19.

(e) Service tunnel.

(f) Vacuum pump cubicle.

(g) Filter pit.

(h) Tank Farm sampler.

(i) Hot can storage shed.

(j) Cubicle "D".

(k) W-19 and W-20 Tank area.

(l) Other radiation zones may be designated on a temporary basis as the need arises.

Table 2 - Procedure for Posting and Establishing Radiation Zones

No.	Dose Rate Range	Immediate Action	Follow-Up Action
1	3 mrem/hr - 6 mrem/hr	Post low level tags if the accumulated daily dose to personnel may be 20 mrem.	Periodic review
2	6 mrem/hr - 1 rem/hr	Post warning signs or tags	Rope off the area if the accumulated weekly dose may be 1 rem.
3	1 - 3 rem/hr	Post warning signs or tags. Rope off.	Erect a barricade which provides absolute physical exclusion if the accumulated weekly dose in the area may be 12 rem. Lock or block entrance.
4	Over 3 rem/hr	Post warning signs, tag, and erect a temporary barricade. Lock and/or block all entries.	

4. Contaminated Vehicles

   A. The bed of the one and one-half ton truck is a contaminated zone. The cab of the truck is a regulated zone.

5. All personnel must be thoroughly checked before leaving the work area in each building. Victoreen meters and hand and feet counters are available in the building for self monitoring. If in doubt, call Health Physics,

Telephone 6263. If the required instrument or instruments are not available regardless of the reason call Health Physics.

6. In areas where hand decontamination facilities are available, the hands must be decontaminated before leaving the work area.

7. If the clothing and/or shoes have any detectable contamination, change to clean clothing and put on shoe covers before leaving the work area. Request the necessary assistance to meet this requirement from your supervisor.

8. Contamination marked clothing may be worn anywhere in the F3P area provided they are free of contamination.

9. Personnel must not wear contamination marked clothing outside the F3P area except:

   A. If free of contamination, the individual may ride in a regulated zone vehicle from one regulated zone to another regulated zone.

10. Personnel working in contamination zones must be clothed in contamination marked coveralls.

11. Smoking and eating are prohibited in contamination zones.

12. The requirements for entry into a radiation zone are in Table 3.

Table 3 - Requirements for Entry into a Radiation Zone

I	II	III	IV	V	VI
			Administrative Authority		
Exposure Range (rem/hr)	Direct Reading Monitoring Instruments Required	Health Physics Surveillance Required	Employee's Division Director	Health Physics Division Director	Laboratory Deputy Director
0.003 - 5[1]	X	X			
5 - 20	X	X	X		
20 - 50	X	X	X	X	
Over 50	X	X	X	X	X

[1] In the exposure range 0.003 - 5 rem/hr, the requirements specified in Columns II and III may be ignored if the anticipated exposure time is such as to result in an accumulated weekly dose of less than 0.1 rem.

13. Trainees, personnel on loan to the Laboratory, and visitors are subject to the provisions of these regulations in the same manner as Laboratory employees.

APPENDIX NO. 7

PROCEDURE FOR PERSONNEL ENTRY TO FISSION PRODUCT PILOT PLANT CELLS

I. Cell Danger Zone

Definition: An area where high level radiation and contamination can be expected or is expected to exist.

F3P Cell Danger Zones

1. Process Cells 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 20, 21, 22, 23, 24, 25 and 26.
2. Manipulator Cells 11, 12, 13, 14, 15, 18, 19 and 27.
3. Decontamination Cell No. 16.
4. Service tunnels.
5. Pipe tunnels.

II. Entry Procedure

Entry to a Danger Zone requires that the following items be completed before personnel shall enter the area:

1. A survey by the F3P Supervision or Health Physics surveyor with the proper equipment for determining personnel exposure.
2. The completion of a Work Permit by the F3P supervision or the Health Physics surveyor.
3. Personnel shall 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 badge.
   (d) Screamers.
   (e) Film rings will be worn on the hand normally used for hand operations.

4. 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 Department Superintendent.

   If a non-plastic suit area requires a gas mask, all clothing will be taped and no part of the body shall be exposed.

5. A timing device shall be set for the determined working time and shall be equipped with a mechanical or electrical alarm.

6. An observer is required for entry into a Danger Zone and he shall be in a location where he can render immediate air or call for assistance if required.

7. A monitor device shall be placed in the Danger Zone with an observer or an alarm unit located outside the area to monitor sudden changes in radiation level.

8. F3P personnel will enter a Danger Zone with a cutie pie in their possession.

9. F3P personnel will enter top access cells with a life line attached to their waist.

III. In-Cell Procedure

1. Enter cell by the shortest but safest route.

2. Do not remove anything by hand. Use tongs, pliers, extended brushes, or any other device which will keep the hands and body in radiation fields less than 5 r/hr.

3. Check all suspicious material with the cutie pie.

4. Check dosimeter periodically and leave immediately if a reading of greater than 100 mr/hr is recorded.

5. Place cutie pie in a position where it can be read at a glance.

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 greater air supply and generally means a departure from safe work habits.

IV. Departure Procedure

Personnel leaving a Danger Zone shall follow the procedure outlined below:

1. When the observer or the alarm signal 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 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.

Temporary Form

ISOTOPES DIVISION WORK PERMIT

Date _____ Shift _____

Operator _____ Badge No. _____
Location _____ Time _____
Survey By _____ Air Count _____
Radiation Reading _____
Instrument Used _____
Working Time _____

Description of Work _____

PROTECTIVE EQUIPMENT REQUIRED:

Clothing		Safety Equipment		Radiation Monitors	
Safety Shoes	_____	Safety Glasses	_____	Pencil Meters	_____
Coveralls	_____	Air Line Suit	_____	Dosimeter	_____
Cap	_____	Assault Mask	_____	Film Badge	_____
Gloves: Cotton	_____			Cutie Pie	_____
Rubber	_____			Film Ring	_____

Observer _____ Time In _____ Time Out _____

WORK COMPLETION

Operator Surveyed By _____
Dosimeter Reading _____
Personnel Decontamination _____

Remarks _____

Foreman _____

TX-2125

APPENDIX NO. 8

F3P EMERGENCY AND EVACUATION PROCEDURE, BUILDING 3517

The attached area emergency and evacuation procedure is approved and will become effective immediately.

Approved by: Original Signed by J. H. Gillette
Division Director

Original Signed by
R. W. Schaich
Area Emergency Supervisor

Original Signed by
T. W. Hungerford
Supt., Safety and Fire Control

BUILDING EMERGENCY AND EVACUATION PROCEDURE

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INSTRUCTIONS FOR ALL NON-EMERGENCY EMPLOYEES

FIRE OR EXPLOSION

Any employee discovering a fire or explosion should actuate the nearest fire alarm box or call the Fire Department emergency number 6359 and give the circumstances of the fire after which the employee should notify or have someone notify the Area Emergency Supervisor and attempt to control the fire at hand if it is practicable to do so.

HAVE SOMEONE MEET FIREFIGHTERS AND DIRECT THEM TO THE SCENE OF FIRE

If it is obvious that the building or area should be evacuated, cause the evacuation alarm to be sounded immediately.

FOLLOW THE EVACUATION PLAN IF THE EVACUATION SIGNAL SOUNDS

NOTE: A small fire (one which the employee is completely sure that he can extinguish) may be extinguished without adherence to the above, after which the Fire Department and Area Emergency Supervisor must be notified.

ANY OTHER EMERGENCY

If the emergency is of such a nature as to require medical assistance, ambulance, patrol, etc., call the Laboratory emergency number 6500 and give the circumstances of the emergency after which the employee should notify the Area Emergency Supervisor and attempt to control or assist in controlling the emergency with the equipment or supplies at hand if it is practicable to do so.

HAVE SOMEONE MEET THE EMERGENCY PERSONNEL AND DIRECT THEM TO THE SCENE

If it is obvious that the area should be evacuated, cause the evacuation signal to be sounded immediately.

FOLLOW THE BUILDING EVACUATION PLAN IF THE EVACUATION SIGNAL SOUNDS

NOTICE:

Any employee hearing the Laboratory evacuation signal which is a series of thirty seconds continuous ringing of the bell system (Note: Not to be confused with the take cover alarm signal which is a series of thirty seconds intermittent ringings of the bell system) sounded simultaneously with rapid successive blasts of the fire horn, indicating that the entire Laboratory is to be evacuated will immediately cause the building evacuation signal to be sounded. If not assigned to regular emergency duties, he will follow the area evacuation plan in carrying out the Laboratory evacuation procedure in accordance with a prearranged plan or as instructed by the Area Emergency Supervisor.

EVACUATION DRILL INSTRUCTIONS

When the evacuation drill signal sounds all employees who are not assigned to perform emergency duties will:

(a) Stop work.

(b) If practical, shut off power. If not practical to shut off power, shut off any electrical machines and turn off lights.

(c) If practical, shut off gas and extinguish any other open flames.

(d) Place classified documents in vaults or file cabinets and lock them.

(e) Close all doors and windows, except those to be used as exits, opening upon or under fire escapes.

(f) Rapidly walk out of building according to building evacuation plan or according to Warden's instructions. Assemble on White Oak Avenue. Return to work area when instructed by Warden.

(1) Don't run

(2) Don't crowd the person ahead

(3) Don't lag behind and thus slow or break the column

(4) Don't scream, laugh, or talk, causing unnecessary noise

(5) Don't cause confusion by becoming unnecessarily excited

(6) Don't remain in toilet or dressing room

(7) Don't return for clothing or personal items

(8) Don't attempt to leave assembly point until dismissed or until it is permissible to return to the building

(9) Don't attempt to leave building except in accordance with evacuation drill regulations

(10) Don't fail to follow instructions

DUTIES OF BUILDING EMERGENCY PERSONNEL DURING ACTUAL EMERGENCIES OR EMERGENCY DRILLS

1. Building Emergency Supervisor

Shift Foreman

He will have charge of the area and will directly combat emergencies in the area until relieved by the Laboratory Shift Supervisor. The Area Emergency Supervisor will coordinate and supervise training of the area combat squad instructed to combat emergencies and to do rescue work in a designated area. He will have general charge of all matters pertaining to evacuation drills and organization; enforce disciplinary measures for failure on the part of the occupants to observe rules or requirements; see that overcrowding in rooms or elsewhere is prevented and that sufficient space is given to aisles and passageways to permit quick access to all of the exits.

(a) He shall prepare and have conspicuously posted on each floor or each building a notice of the schedule of evacuation and the duties of occupants in case of an alarm.

(b) He shall make a survey of the building to determine the capacity of all exit facilities in accordance with the requirements of the Safety and Fire Control Departments.

(c) He shall assign exits for the occupants of each room.

(d) When changes are made in the occupancy conditions he shall make such changes in the assignment of exits as may be necessary.

(e) He will notify all members of the evacuation drill organization regarding the general plan of exit assignment and the details pertinent to their specific duties, and examine them orally as to their familiarity therewith, going over the ground when necessary.

(f) He shall be responsible for planning the shutting off of electricity, processes, gas, oil, etc., in case of alarm or fire.

(g) He shall personally supervise the sounding of the general building alarm and see that each movement corresponding to alarm signal is promptly and properly executed.

(h) He shall be responsible for the condition of all aisles and passageways and shall see that chairs, benches and stock in transit are promptly removed to insure unobstructed passageways.

(i) He shall select and designate the exits to be used by the occupants on his floor.

(j) It is required that he instruct the occupants in the use of all means of egress so that they will be familar with all routes.

2. Alternate Building Emergency Supervisor

R. W. Schaich

He shall assume all the duties of the regular Building Emergency Supervisor in his absence.

3. Searchers

Searchers will be assigned by the Wardens at the time evacuation alarm is sounded. If searchers are not available, the Emergency Supervisor himself will perform the duties of the searchers.

The Searchers will check all rooms in their territory to be sure that evacuation is complete and that all doors and windows are closed. They shall visit the toilet rooms and any rooms frequented by their sex. They shall look out for any people who may become hysterical and faint.

4. Combat Squad Members

D. Hodges	E. Cagle
E. Ryan	D. King

These combat squad members are instructed to combat fire with equipment at hand in the area or building and are trained to combat other emergencies. Upon receiving notification of any emergency they report to the Area Emergency Supervisor and combat the emergency in accordance with his instructions or according to a prearranged plan.

Approved by: Original Signed by E. E. Beauchamp

APPENDIX No. 9

F3P PROCESS DATA

Batch No. _____ Tank No. _____
Previous Code _____

Crystallization

1. Time Started _____ Date _____
2. Chemicals Added:
   _____ lbs. Na_2SO_4 _____ Gals. Water
   _____ lbs. $(NH_4)_2SO_4$ _____ Gals. 60% HNO_3
   _____ lbs. Alum _____
3. Temperature Break _____ °C
4. Final Temperature _____ °C
5. Crystal Volume _____ Gals.
6. Crystal Bed Disposition _____
7. Time Completed _____
8. Operator _____
9. Shift Change Status _____

Precipitation

1. Time Started _____ Date _____
2. Chemicals Added
   _____ Gals. 19 M NaOH _____ Cubic Feed NH_3
   _____ Liters 42-1/2% Hydrazine _____ Lbs. Oxalic Acid
   _____ Lbs. Na_2CO_3 _____ Grams $Y(NO_3)_3$
   _____ Grams $Fe(NO_3)_3$ _____ Gals. 60% HNO_3
   _____ Liters H_2O_2 _____ Liters NH_4OH
   _____ Di Sodium Phosphate
   _____ _____
3. Digestions:
   (a) Temp. _____ °C Time _____ Hrs. Time End _____
   (b) Temp. _____ °C Time _____ Hrs. Time End _____
   (c) Temp. _____ °C Time _____ Hrs. Time End _____
4. Final pH or Normality _____
5. Precipitate Volume _____ Gals.
6. Centrifugation Rate _____ G.P.M.
7. Batches in Centrifuge Bowl _____
8. Dissolved Precipitate to Tank _____
9. Time Completed _____
10. Operator _____
11. Shift Change Status _____

SAMPLE DATA

Starting Solution

Sample Code _____ Tank No. _____
Liquid Level _____ Gamma Reading _____
Specific Gravity _____ Operator _____
Volume _____ Gals. Date _____

Lab Centrifuge

Sample Codes _____ A _____ B Tank No. _____
Liquid Level _____ Operator _____
Specific Gravity _____ Date _____
Volume _____ Gals.

Waste Solution

Sample Code _____ Tank No. _____
Liquid Level _____ Gamma Reading _____
Specific Gravity _____ Operator _____
Volume _____ Date _____

CESIUM PURIFICATION

Batch Data Card

Batch No. Date _____

Operator _____

1 and 2. Operational Checks

3. Transfer

Transfer No.	Volume Transferred	C-513 Crystal Volume	Operator
1			
2			
3			
4			
5			

4. Crystallization

Water Crystallization No.	C-513 Crystal Volume	Operator
1		
2		
3		
4		

5. Rubidium Removal

Removal No.	Alum Added	Crystal Volume	Time	Operator
1				
2				
3				
4				
4				
5				
6				
7				

Water Rinse

Volume H_2O Added _____ Time _____ Operator _____

6. Cesium Dissolution

NaOH Scrubber Setting _____ E-213 Off Gas _____

HCl Added _____ Liters Time _____ Operator _____

7. Cesium Precipitation

b. Recovery Pt Added _____ Grams Date _____

New Pt Added _____ Grams Time _____

Total Pt Added _____ Grams Operator _____

	Process	Temperature °C	Time Start	Time Complete	Operator
c.	Digestion				
e.	Settling				

Transfer and Rinse Data:

	Process	Volume	Final Disposition	Time	Operator
f.	Filtration				
g.	First HCl Rinse				
h.	Second HCl Rinse				
i.	Third HCl Rinse				
j.	H_2O Rinse				
m.	Sample				

8. Cesium Chloroplatinate Dissolving

a. 1 N HCl Added _____ Liters Date _____

d. 42.5% Hydrazine Added _____ Liters Operator _____

Process	Temperature °C	Time Started	Time Completed	Operator
f. Digestion				
g. Digestion				

9. Cesium Evaporation

a. NaOH Scrubber Flow _____ Operator _____

b. E-213 Condenser Water _____ E-213 Off Gas _____ Operator _____

Process	E-213 Starting Volume	E-213 Final Volume	Steam Pressure	Operator	Time
c. Filtration					
h. HCl Rinse					
i. Vol. Reduction					
j. HCl Addition					

10. Ammonium Salt Destruction

Process	Volume Acid Added	E-213 Volume	Steam Pressure	Time	Operator
c. Aqua regia					
e. Aqua regia					
g. Aqua regia					
i. Aqua regia					
k. Aqua regia					
l. Vol.reduction					

10. Ammonium Salt Destruction (cont'd)

Process	Volume Acid Added	C-213 Volume	Steam Pressure	Time	Operator
m. HCl Addition					
o. Aqua regia					
q. Aqua regia					
s. Aqua regia					
u. Aqua regia					
w. Aqua regia					
x. Vol. reduction					

11. Acid Removal

Process	Volume Acid Added	E-213 Vol. Start	E-213 Vol. Finish	Steam Pressure	Time	Operator
a. HNO_3 reduction						
c. HNO_3 reduction						
e. HCl reduction						
g. HCl reduction						
i. HCl reduction						

12. Powdering

a. Tray Clean and Drain Plugged Operator _____

Process	Volume to Beaker	Time Start	Time Complete	Operator
d. Filtration				
h. First drying				
h. Second drying				
j. Sampling				

Process	Wt./grams	Can No.	Storage No.	Time	Operator
k. Weighing					
Weighing					
Weighing					
Weighing					
Total Weight					

13. Platinum Recovery

	Process	Solution Added	Temperature °C	Time	Operator
c.	Pt. Ppt.				
d.	Digestion				
g.	Filtration				
h.	Rinse No. 1				
	Rinse No. 2				
	Rinse No. 3				
j.	Pt Dissolving				
l.	Digestion				

	Process	E-213 Start	E-213 Finish	Time	Operator
o.	Vol. Reduction				
r.	Sampling				

PRECIPITATION

Operational Instructions

Date _____ Supervisor _____

Chemicals

		Operators Initials
1.	_____ gals. 70% HNO_3 per batch	_____
2.	_____ gals. 4 M Na H_2PO_2 per batch	_____
3.	_____ gals. 50% NaOH per batch	_____
4.	_____ liters hydrazine per batch	_____
5.	_____ cu. ft. NH_3 per batch	_____
6.	_____ grams Na_2CO_3 per batch	_____
7.	_____ grams $Fe(NO_3)_3$ per batch	_____
8.	_____	

Samples

Tank No.	Code	Dilution	Sample Volume	Disposition
P1	_____	_____	_____	_____
P2	_____	_____	_____	_____
P3	_____	_____	_____	_____
P4	_____	_____	_____	_____
H4	_____	_____	_____	_____

Precipitator Disposition

Tank	Volume	Process	Centrifugate	Precipitate	Dissolved Precipitate
P-12	_____	_____	_____	_____	_____
P-23	_____	_____	_____	_____	_____
P-34	_____	_____	_____	_____	_____
P-45	_____	_____	_____	_____	_____

Special Instructions

CRYSTALLIZATION

Operational Instructions

Date _____ Supervisor _____

Chemicals		Operators Initials
1. _____	lbs. Na_2SO_4 per feed batch	_____
2. _____	lbs. $(NH_4)_2SO_4$ feed batch	_____
3. _____	lbs. alum per feed batch	_____
4. _____	gals. water per feed batch	_____
5. _____	gals. 70% HNO_3 per feed batch	_____
6. _____		_____

Samples

Tank No.	Code	Dilution	Sample Volume	Disposition
C-1	_____	_____	_____	_____
C-2	_____	_____	_____	_____
C-3	_____	_____	_____	_____
C-4	_____	_____	_____	_____
S-3	_____	_____	_____	_____

Crystallizer Disposition

Tank	Volume	Filtrate	Crystal Bed
C-17	_____	_____	_____
C-27	_____	_____	_____
C-38	_____	_____	_____
C-48	_____	_____	_____

Special Instructions

APPENDIX No. 10

F3P DECONTAMINATION PROCEDURE

I. Cell Equipment Decontamination

1. Secure all jet switches going to tanks that contain radioactive solutions. If additional safety is required, have Instruments 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. The following chemicals will be added to the starting vessels and will be heated to 90°C for 15 minutes, cooled and jetted 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 oxalic.

   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 H.P. 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 chemicals:

   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

NOTE: Keep sprays at a distance from centrifuge motors. Do not operate agitators or centrifuges while spraying.

APPENDIX NO. 11

F3P SHIFT TANK INVENTORY

Date _____
Shift _____
Operator _____

Tank No.	Batch Nos.	Volume Percent	Remarks
F-421			
F-521			
F-621			
F-821			
F-1021			
F-1121			
W-19			
W-20			
S-122			
S-324			
S-223			
S-523			
S-424			
P-12			
G-12			
H-12			
F-12			
P-23			
G-23			
H-23			
F-23			
P-34			
G-34			
H-34			
F-34			
P-45			
G-45			
H-45			
E-16			
C-17			
C-27			
C-38			
C-48			
P-511			
H-511			
R-111			
P-89			
H-89			
N-19			
N-29			
R-29			
R-39			
R-79			
C-513			

Shift Tank Inventory (Cont'd)

Tank No.	Batch Nos.	Volume Percent	Remarks
C-613			
E-213			
FV1			
FV2			
M-833			

APPENDIX NO. 12

Operator _____

Date _____

BUILDING 3517 CHECK SHEET

1. Jet the following sumps to the S-223 tank:

   (a) Cell 21 _____

   (b) Cell 22 _____

   (c) Cell 23 _____

   (d) Cell 24 _____

   (e) Cell 25 _____

   (f) Cell 26 _____

   (g) Jet W-19 and W-20 valve pit to W-12.

2. Distilled water system check.

   (a) Distilled water tank volume _____

   (b) Steam pressure _____

   (c) Water on condenser _____

   (d) Pump operation _____

3. Air dryer check.

   (a) Air pressure _____

   (b) North unit temperature _____

   (c) South unit temperature _____

   (d) Rotometer free of moisture _____

4. Off gas.

   (a) Process cell _____ "H_2O

   (b) Manipulator cell _____ "H_2O

5. Cell ventilation.

   (a) Cell No. 1 _____ "H_2O

   (b) Cell No. 13 _____ "H_2O

   (c) Filter manometers North CWS_____"H_2O South FG____ "H_2O

   (d) Steam on to jets North_____ South _____

6. Vacuum pump.

   (a) Pump operation _____

   (b) Vacuum _____ "Hg

   (c) Rotometer setting _____

   (d) Suck H_2O from pan _____

7. Chilled water unit. On_____ Off_____

   (a) Chilled water temperature Outlet_____°C Inlet_____°C

   (b) Chilled water pump operation _____

   (c) Freon Suction_____Discharge_____

   (d) Tower level _____

8. Lights.

   (a) Exit _____

   (b) Outside building _____

   (c) Inside building _____

   (d) Emergency shower _____

9. Hot can storage.

   (a) Empty _____

   (b) Full _____

9. (Cont'd)

Weekly Checks on Friday

(a) Emergency lanterns _____

(b) Emergency siren _____

(c) Entrance doors lock system _____

(d) Emergency showers _____

(e) Gas masks boxes are supplied _____

APPENDIX NO. 13

CELL 14 STORAGE WELL OPERATION PROCEDURE

1. To fill storage well with water, open distilled water valve located on southeast wall of Cell 14 to well to be filled.

2. The liquid level instrument is calibrated from 0 to 10 inches of water. The high or amber light is 1/2 inch below the overflow to S-523. The normal or green light is two inches below overflow. The low or red light is three inches below overflow.

3. Maintain a green light or normal level when products of powder or pellets are stored in wells.

4. The cooling coil water to storage well located on panelboard should be set at approximately 25% on flow meter to hold temperature at approximately 20 to 30°C.

5. Set air sparge flow meter at 20% on panelboard. The air sparge is to circulate the water around cans and cooling coils.

6. To empty storage well water to S-523, open siphon water valve located on southeast face of Cell 14 until line is primed and close valve.

APPENDIX NO. 14

F3P CHEMICAL INVENTORY

Date _____
Shift _____
Operator _____

Make-Up Tank Chemicals

Tank No.	Chemical	Volume Gallons or Liters	Remarks
M-131	19.0 M NaOH		
M-231	19.0 M NaOH		
M-331	60% HNO_3		
M-431	C.P. 19 M NaOH		
M-1			
M-2			
M-532	10 N HNO_3		
M-632	60% HNO_3		
M-732	2 N HNO_3		
M-1034	19 M NaOH		
M-1134	60% HNO_3		

Supply Chemicals

Name	Container	Quantity	Max.	Min.	Remarks
60% HNO_3	Carboy		30	20	
70% HNO_3	Carboy		3	1	
70% HNO_3	Case		3	1	
NaOH C.P. Pellets	100 lb. carton				
NaOH (flake) Rayon	100 lb. drum		80	60	
$(NH_4)_2SO_4$ R.G.	100 lb. drum		40	15	
NaH_2PO_2 C.P.	50 lb. drum				
Hydrazine	55 gal. drum		15	10	
Alum	100 lb. bag				
Na_2SO_4	100 lb. bag		85	42	
$Fe(NO_3)_3 \cdot 9H_2O$	50 lb. drum		4	2	
Versene	25 lb. carton		4	2	
pH 2	Gal. bottles		5	2	
pH 4	Gal. bottles		5	2	
pH 7	Gal. bottles		5	2	
pH 10	Gal. bottles		5	2	
Citric	100 lb. drum		2	1	
Amsco 125-82 (Super Sol)	55 gal. drum		4	2	
D_2 Ethyl Hexyl Phosphoric Acid	55 gal. drum				
Hydrogen Peroxide C.P.	13 gal. carboy		2	1	

Supply Chemicals (Cont'd)

Name	Container	Quantity	Max.	Min.	Remarks
$KMnO_4$	5 lb. bottles		10	4	
HCl	Case		4	2	
NH_3 Cylinders	Cylinders		4	3	
Oxalic Acid	300 lb. drum		2	1	
Soda Ash	100 lb. bag		3	1	
Cerium Nitrate	150 lb. drum		1	0.5	
NH_4OH	Case.		3	1	
H_2PtCl_6	1 lb. bottles		2	1	

APPENDIX No. 15

F3P SAFETY REGULATIONS, BUILDING 3517

1. Protective Clothing and Equipment

(a) Safety glasses, safety shoes, film badges and pocket meters will be worn at all times in all areas, except while showering.

(b) Face shields, rubber gloves and arm covering will be worn whenever chemicals are handled or transferred by pump.

(c) Hard hats will be worn whenever it is necessary to enter a cell or pipe tunnel when the roof blocks are removed.

2. Handling and Storing of Chemicals and Equipment

(a) All materials weighing over 50 pounds will be handled by two or more men or the crane. The weight limit is 50 pounds per man.

(b) Loading areas will be chained off when lifting equipment or chemicals with the crane.

(c) Gas cylinders will be handled according to standard procedure and all empties will be tagged and placed in the empty racks.

(d) Carboys will be handled by carboy lifters and drum holders and will be flushed and tagged after emptying.

(e) Acid bottles will be carried in approved containers and all empties cleaned immediately after emptying.

(f) All empty chemical containers shall be removed from the building and stored in proper places as soon as possible.

(g) Hydrazine will be less than 42.5% concentration and will be stored in approved location.

(h) Hydrazine will be handled in the same approved manner as in the chemical make-up procedure.

(i) No solvents will be stored inside the building.

(j) Solvents will be transferred in approved safety cans with grounding clamps.

(k) Flammable material will not be stored on stairway storage areas.

(l) All areas shall be kept clear of material at the safety showers, fire fighting equipment and exits.

3. Mechanical and Electrical Equipment

(a) No mechanical equipment such as cranes, pumps, centrifuges, agitators, etc., will be operated until the operator personally clears the area of personnel.

(b) Main electrical switches will be turned off and tagged whenever work is necessary on electrical equipment.

(c) Danger tags will not be removed from equipment by anyone except the person who originally placed it on the equipment.

(d) When not in use, the bridge crane will always be parked over the loading bay with the crane hook at least ten feet above the floor.

4. Fire Fighting and Emergency Equipment

(a) All personnel shall acquaint themselves with operation and location of all fire fighting and other emergency equipment.

(b) All personnel shall acquaint themselves with the operation and location of all safety showers.

(c) All personnel shall acquaint themselves with the operation and location of assault and air line masks.