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PROCESS SPECIFICATIONS
FOR OPERATIONAL CONTROL - PUREX PLANT
(REVISION NO. 1)

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PUREX PLANT (REVISION NO. 1)

By

E. R. Irish

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Research and Engineering
CHEMICAL PROCESSING DEPARTMENT

November 17, 1958

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PROCESS SPECIFICATIONS FOR OPERATIONAL CONTROL -
PUREX PLANT (REVISION NO. 1)

3.01 Introduction

The Process Specifications for Operational Control of the Purex Plant⁽¹⁾ have been revised to define the operational control changes as a result of (a) converting the Purex Plant from three to two cycles, and (b) the use of the Plutonium Anion Exchange Unit (N-Cell) for routine plutonium concentration in place of the L-Cell Concentration Package. The flowsheets specified in Figures I-1, -2, and -3, and Table I fulfill the requirements for nuclear and chemical safety within the process as defined in Purex Process Specifications 1.00⁽⁷⁾ and 2.00⁽²⁾. The flowsheet also provides adequate recovery and decontamination to meet the product purity requirements itemized in Section 3.04. To clarify the two-cycle flowsheet and to simplify its use, the specifications are listed on graphic flow diagrams with the notation that process control allows a deviation of plus or minus five percent on all specifications except TBP concentration in solvent.

3.02 Revisions

The flowsheet conditions may be changed within the following limitations:

- (a) New flowsheet conditions will fulfill the requirements of the nuclear safety and chemical hazards specifications.
- (b) New flowsheet conditions require the approval of the Manager, Purex Technology Operation.

3.03 Purex Plant Flowsheet (PPFS IV)⁽¹⁾⁽³⁾⁽⁵⁾

The flowsheets presented in Figures I-1, -2, and -3, and Table I are based on operation with greater than 400 MWD/T metal. Flow ratios are relative to 75 flows of HAF at 1.8 M (3.57 lb./gal.) uranium concentration. (HAF flow = 0.388 gpm/ton/day, plus 0.2 gpm HS pulser leakage which is recycled to the HAF Tank.) Temporary deviations which are greater than the process control limits are permissible during approved test operations or when overall rates are being changed or during start-ups and shutdowns. However, efforts should be made to attain the steady-state values within the minimum practicable time.

3.03.01 Coating Removal and Metal Dissolution

The Coating Removal and Metal Dissolution process specifications are shown in Figure I-1, along with the Iodine Backup Facility.

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Coating Removal is based on a nine-ton charge of standard irradiated, natural uranium slugs. Metal Dissolution is based on two 4-1/2 ton cuts. The endpoint sp. gr. for dissolution is 1.75 hot. The off-gas leaving the dissolver tower must be below 50° C, and entering the silver reactor above 190° C.

3.03.02 Solvent Extraction Flowsheet IV

The solvent extraction cycles are shown in Figures I-1, -2, -3 and Table I. The steady-state values are based on controlled product losses in the 2DW, 2AW, and XAW waste streams which are backcycled to the 3WB stream for ultimate recovery in the HA Column. The desired control limits for backcycle losses are:

2DW - 5 ± 2 percent uranium loss
 2AW - 1 ± 0.5 percent plutonium loss
 XAW - 10, or less, percent plutonium loss

The recycle of product in the IBSU stream to the HA Column via the HAO stream is estimated to be 2 and 8 percent for uranium and plutonium, respectively.

3.03.03 Plutonium Concentration Flowsheet IV & IV-A

The final plutonium concentration is routinely performed in the Arion Exchange Unit as shown in Figure I-2; however, an alternate concentration method is available. Plutonium Concentration Flowsheet IV-A, utilizing the L-Cell Concentration Package, is listed in Table I. The principal difference in these flowsheets is the sulfate concentration in the 2BX stream, which is 0.045 M H₂SO₄ for N-Cell operation and 0.003 M H₂SO₄ for L-Cell operation. For nitric acid concentration limits which meet product specifications and prevent plutonium polymer formation, see Figures II and III.

3.03.04 Organic Treatment Flowsheet IV

The Organic Treatment Flowsheet IV is shown in Figure I-3. All flow values are shown as continuous flows; however, the IOBW, IORW, and 2ORW are changed on a batch basis of 1000 gallons at one time.

3.03.05 Waste Treatment and Acid Recovery Flowsheet IV

The Waste Treatment and Acid Recovery Flowsheet IV shown in Figure I-3 is based on the following conditions:

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- (a) The Acid Evaporator does not overflow to the IWF Tank. Periodic removal of the 2WW bottoms will be performed during plant shutdowns.
- (b) The reflux water ratio AAR to AAD in the Acid Absorber is 0.25.
- (c) The recovery of acid from the Iodine Backup Facility is 70 ± 10 percent.
- (d) The addition of UO_3 recovered acid to the AFF is 2.5 parts UO_3 acid to 10 parts AAA plus Backup Facility acid on a nitric acid weight basis.
- (e) The reflux water ratio AFR to AFD in the Vacuum Fractionator is 0.35.

Flowsheet compositions of IWW, neutralized waste, and self-concentrated waste are average values based on material balances and analytical analyses.⁽⁸⁾

3.04 Product Specifications

The specifications for product purities are dependent upon the requirements of the facilities receiving these products and, hence, are not established by the Purex Technology Operation. However, product purity specifications are tabulated below for reference purposes since they represent one of the major goals of flowsheet specifications. Solutions which do not quite meet these specifications may be shipped, if approved on a waiver basis by the Finished Products Technology Operation.

3.04.01 Uranium Specifications⁽¹¹⁾

Uranium Concentration:	4.0 to 4.4 lb./gal. (1.64 to 1.72 Sp. Gr.)
HNO_3 Concentration:	Less than 0.1 lb. HNO_3 /lb. U
Plutonium Concentration:	Less than ten parts per billion parts of U.
Total Metallic Impurities: (Na, Cr, Fe, Ca, Ni, Al, Mo)	Less than 200 parts per million parts of U. (Product with 200 to 500 parts per million parts of U may be shipped upon approval.)

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Fission Products Beta Activity: Less than 100 percent of the beta activity of aged, natural U.

Total Gamma Ratio (GR)*: Less than 2.0. (Shipments of product greater than 2.0 are subject to negotiation.)

* As reported by laboratory and includes uranium daughters.

3.04.02 Plutonium Specifications (6)(7)(9)(10)

Plutonium Concentration: Greater than 130 grams per liter with a suggested upper limit of 270 grams per liter.

HNO₃ Concentration: Ideal range 5.0 to 7.0 M. However, the acidity is variable depending on the volume in the PR Can, as shown in Figure II.

PR Can Volume: 5.3 to 9.0 liters. See Figure II.

Total Pu per PR Can: 700 - 2000 grams.

Uranium: Shall not exceed 0.5 percent by weight of the plutonium.

$$\frac{U \text{ lb./gal.}}{AT \text{ c/m/gal.}} = \angle 1.5 \times 10^{-16}$$

Metallic Impurities: The sum of Fe, Cr, Ni, and Al shall not exceed 200,000 ppm of Pu. Neither Fe nor Al shall exceed 100,000 ppm of Pu.

Gamma Activity:

The G/AT ratio in this specification is defined as the ratio of total fission product gamma (microcuries/gallon) to total plutonium alpha (counts/minute/gallon).

1. G/AT less than 3.5×10^{-11}

Acceptable at Z Plant without waiver.

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2. G/AT between 3.5×10^{-11} and 4.0×10^{-11}

A waiver is required from Z Plant prior to acceptance. If only one batch is involved (6 - 8 cans), no blending at Z Plant will be required. If more than one batch is in this range, blending in Z Plant will be scheduled.

3. G/AT between 4.1×10^{-11} and 6.0×10^{-11}

A waiver is required from Z Plant prior to acceptance. Acceptance will be recommended if there is material on hand at Z Plant for blending in a ratio of at least 1:1. The material used for blending should have a G/AT of 3.0×10^{-11} , or less. (Zr-Ru ratio information would be helpful in scheduling the blending of this material.)

4. G/AT between 6.1×10^{-11} and 8.0×10^{-11}

A waiver is required from Z Plant prior to acceptance. Batches in this range will only be considered for acceptance if the Zr-Nb ratio is less than 0.4 and there is material on hand at Z Plant for blending in a ratio of at least 1:1.

3.05 Process Critical Mass Control Limits⁽⁷⁾

Critical mass control in the Purex Plant is based on the document, "Process Specifications for Critical Mass Control - Purex Plant, (Revision No. 2)".⁽⁷⁾ Presented below are the process vessel control limits based on the specifications presented in the reference document. In addition, pertinent control limits for the handling of recycle solution and resin handling have been quoted. In the case of additional questions concerning critical mass control, the reference document should be consulted.

3.05.01 Process Vessel Control Limits

Process equipment control limits are listed in Table II. Nuclear safety is the primary guide used in establishing maximum process control limits in process vessels. For the solvent extraction and feed preparation vessels, the maximum limits are based on a critical mass control limit of 1900 grams of plutonium in the 2AF TK-J5. During normal process operation, the plutonium concentration in some vessels is far below the nuclear safety limit, but for process control, the concentration is limited by waste or product specifications. In such cases, waste and product specifications have been used to establish the maximum process control limits. For clarification, Table II shows the 'Control Basis' used for each tank, concentrator, column or sump.

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3.05.02 Recycle Control Limits

The maximum allowable plutonium holdup in an RC Can shall be:

350 grams of less than 400 MWD/T

400 grams of greater than 400 MWD/T

The maximum allowable plutonium holdup in the recycle storage tank shall be 650 grams. Addition to the recycle storage tank of plutonium solutions greater than 13 g/l in concentration shall be restricted by:

- (a) Liquid level in the tank must be less than one inch prior to addition.
- (b) Following addition, the tank contents must be diluted with water or dilute nitric acid while agitating, to less than 13 g/l. See Figure III.

3.05.03 Resin Removal

The handling of plutonium-contaminated resin will be covered by special procedures issued as necessary by the Manager, Purex Technology Operation. No unstripped resin shall be removed from equipment.

3.06 Solvent Extraction Column Data

Specific data for the solvent extraction columns are listed in Table III.

3.07 241-A Tank Farm Operating Control Limits

- 1. Maximum pressure generated within the tank - 10 psig

Note: At the present time the maximum pressure generated is controlled by limiting the depth of the liquid within the tank to a maximum pressure of 10 psig on the tank bottom.

- 2. Maximum storage tank weight factor - 277 inches
- 3. Maximum sodium ion concentration (calculated) - 8.0 M
- 4. Maximum density of stored waste (at boiling temp.) - 1.37 g/cc

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5. Maximum sludge temperature - 300° F

Note: The temperature of the sludge will normally remain less than 300° F; however, during tests or periods of abnormal operation the temperature may exceed 300° F. When this happens, the temperature should be lowered as rapidly as possible by dilution of the tank contents with water accompanied by increased air flow to the recirculators.

6. Maximum cooling water discharge temperature - 158° F

7. Minimum condenser cooling water flow - for E-411 - 100 gpm
- for E-410 - 80 gpm

8. At least one recirculator to be operated as long as mixing of tank contents is required.

9. Air rate to each recirculator - 10 cfm

10. Tanks to be put into service require at least 60 days to heat from ambient to boiling with a maximum temperature increment of 80° F allowed.

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TABLE I

PLUTONIUM CONCENTRATION FLOWSHEET IV-A

Stream	Relative Flow	Sp. Gr. 25° C	Temp. °C	Composition		
				Pu, g/l	HNO ₃ , <u>M</u>	H ₂ SO ₄ , <u>M</u>
2BX	4.7	1.00	30 ± 5	-	0.15	0.003
2BP	4.75	1.02	Ambient	3.80	0.365	0.003
2BP-HNO ₃	0.17	1.37	Ambient	-	13.0	-
PSF	4.92	1.03	Ambient	3.66	0.80 ^(a)	0.003
PSC	4.7	1.03	Ambient	3.83	0.84	0.003
PSS	3.0	-	100	Live Steam	-	-
PSD	3.22	1.00	< 50	-	Trace	-
LPD	4.62	1.02	< 50	-	0.76	-
IPC	0.08	1.47	110	225 ± 25	5.40	0.175

(a) $\frac{\text{M HNO}_3}{\text{g/l Pu}}$ must be ≥ 0.125 to prevent plutonium polymer formation in the stripper.⁽⁶⁾

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TABLE II

PROCESS VESSEL CONTROL LIMITS⁽⁴⁾⁽⁷⁾⁽¹⁰⁾⁽¹¹⁾

<u>Vessel</u>	<u>Control Basis</u>		<u>Maximum Process</u>
	<u>Nuclear Safety</u>	<u>Process Control</u>	<u>Control Limits</u> <u>AT (Alpha counts/min./gal.)</u>
Tank:			
D1		(b)	9.0×10^8
D2		(b)	9.0×10^8
D3	(a)		2.5×10^{11}
D4	(a)		2.5×10^{11}
D5	(a)		2.5×10^{11}
E1		(b)	3.1×10^9
E3	(a)		2.3×10^{11}
E3A	Max.		3.6×10^{12}
E5	(a)		2.2×10^{11}
E6	(a)		$1.9 \times 10^{11}(f)$
H1	(a)		$1.9 \times 10^{11}(f)$
J1	(a)		$6.6 \times 10^{10}(f)$
J2	Max.		8.6×10^{10}
J3	(a)		4.1×10^{10}
J5	Max.		6.0×10^{11}
L6	Max.		1.3×10^{14}
L9	Max.		1.3×10^{14}
L10	Max.		1.3×10^{14}
L13	Max.		1.3×10^{14}
N1	Max.		1.3×10^{14}
N7	Max.		1.3×10^{14}
K1		(c)	1.0×10^9
K5		(c)	1.4×10^6
K6		(c)	1.4×10^6
F3		(d)	-----
F7		(b)	6.4×10^7
F8	Max.		1.0×10^{11}
F10	Max.		1.0×10^{11}
F12		(b)	5.0×10^9
F13	Max.		1.0×10^{11}
F15		(b)	3.7×10^9
F16		(b)	3.7×10^9
F18	Max.		1.0×10^{11}
G1		(b)	1.2×10^8
G2		(b)	8.8×10^8
F5-7		(b)	8.8×10^8
G8		(b)	8.8×10^8

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TABLE II, cont.

<u>Vessel</u>	<u>Control Basis</u>		<u>Maximum Process</u>
	<u>Nuclear Safety</u>	<u>Process Control</u>	<u>Control Limits</u>
			<u>AT (Alpha counts/min./gal.)</u>
Tank:			
R1		(b)	3.0×10^7
R2		(b)	7.9×10^8
R5-7		(c)	2.7×10^5
R8		(b)	7.9×10^8
U1		(d)	-----
U2		(d)	-----
U3		(d)	-----
U4		(d)	-----
U5		(d)	-----
U6		(d)	-----
M1	Max.		4.3×10^8
Concentrators:			
H4	(a)		7.8×10^9
F5		(d)	-----
F6		(d)	-----
F11		(b)	3.7×10^9
L3	Max.		1.3×10^{14}
L4	Max.		1.3×10^{14}
N6	Max.		1.3×10^{14}
Columns:			
IBX	(a)		8.4×10^{11}
IBS	(a)		6.7×10^{11}
Sumps:			
S-LA	Max.		1.4×10^{14}
S-L, B, C, D	Max.		1.4×10^{14}
S-N			One inch of liquid
PR Can:	(e)		1.3×10^{14}

Notes for TABLE II:

- (a) The control limit for this vessel is not the Maximum Limit allowed by nuclear safety, but is the maximum allowed in order to insure nuclear safety in the 2AF TK-J5 during continuous process operation. See Note (e).

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- (b) The control limit for this vessel is based on process waste control limits.⁽⁴⁾ See Note (e).
- (c) The control limit for this vessel is based on uranium product specifications.⁽¹¹⁾ See Note (e).
- (d) This vessel should not contain detectable amounts of plutonium during normal process operation. See Note (e).
- (e) Maximum nuclear safety limits for individual vessels during non-routine operation are given in "Process Specifications for Critical Mass Control - Purex Plant."⁽⁷⁾
- (f) The maximum process control limits in HAF TK-H1 and 3WB TK-J1 are variable to insure nuclear safety in the 2AF TK-J5. See Figure IV.

TABLE III

SOLVENT EXTRACTION COLUMNS DATA

Column	Plate or Packed Section		Cartridge Details					Louver Plates		Pulser		Phase	
	Height, ft.	I.D. in.	Plate Type	Plate Mat'l.	Hole Size in. Dia.	Free Area %	Plate Spacing in.	No.	Free Area %	Ampl., in.	Freq. Range c/m	Continuous	Pulsed
HA Ex. Scrub	13.9	24	Nozzle	ss	3/16	23	2	4	23	1.1	35-110	Org.	Org.
	19.2	32	Sieve	(a)	(a)	(a)	1	4	16	0.6			
2D Ex. Scrub	14.0	24	Nozzle	ss	3/16	23	2	4	23	1.1	35-110	Org.	Org.
	13.2	32	Sieve	(b)	(b)	(b)	1	3	16	0.6			
HS	18.6	34	Sieve	(a)	(a)	(a)	1	4	16	0.53	35-100	Org.	Aq.
IC, 2E	18.0	34	Nozzle	ss	1/8	10	4	--	--	0.53	35-110	Aq.	Aq.
IBX	28.0	27	Sieve	ss	3/16	33	4	6	16.5	0.84	35-110	Aq.	Org.
IBS	13.3	8	Sieve	ss	1/8	23	2	6	20	0.84	25-110	Aq.	Aq.
2A Ex. Scrub	20.9	7	(c)	(c)	--	(c)	(c)	--	--	1.1	25-110	Org.	Aq.
	9.8												
2B	21.0	7	Sieve	ss	1/8	23	2	--	--	1.1	25-110	Aq.	Aq.
IO, 20	26.3	34	Sieve	Fluoro- thene	3/16	23	4	--	--	0.53	35-110	Org.	Aq.

(a) Cartridge composed of 21% free area stainless steel plates and 23% free area linear polyethylene plates, grouped alternately four stainless steel plates and two polyethylene plates. Hole diameters in stainless steel plates = 0.085 in.; polyethylene plates = 0.1875 in.

(b) Cartridge composed of alternate 21% free area stainless steel plates and 23% fluoroethene plates. Hole diameters in stainless steel plates = 0.085 in.; fluoroethene plates = 0.1875 in.

(c) Packed with 1 in. fluoroethene Raschig rings.

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NOTES:

1. All flows are relative to 100 flows equals 747 gallons/ton uranium.
2. All temperatures are ambient except as otherwise noted.
3. Composition of dissolver reaction gas has been averaged. Composition of gas will vary with time and mode of dissolver operation.
4. Specific gravities are given for all solutions at 25°C, regardless of operating temperature.
5. Solvent refers to 30 ± 1 vol. per cent TBP in a hydrocarbon diluent of specific gravity equal to 0.801.
6. Plutonium concentrations are based on uranium irradiated to 500 g Pu/ton U.
7. Process control allows ± 5% deviation on all specifications except TBP (See note 5).
8. NaNO₂ addition made only when plant Pu product is recycled.

FIGURE I-1
PUREX TWO-CYCLE
FLOWSHEET PPFS IV
IODINE BACKUP
FEED PREPARATION
FIRST DECONTAMINATION
AND PARTITION CYCLE

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NOTES:

1. All flows are relative to 100 flows equals 747 gallons/ton uranium.
2. All temperatures are ambient except as otherwise noted.
3. Specific gravities are given for all solutions at 25°C, regardless of operating temperature.
4. Solvent refers to 30 ± 1 vol. per cent TRP in a hydrocarbon diluent of specific gravity equal to 0.801.
5. Plutonium concentrations are based on uranium irradiated to 500 g Pu/ton U.
6. Anion exchange resin is Permutit SK, 20 - 40 mesh. Flow volumes of resin are for wet settled resin in the nitrate form.
7. Process control allows ± 5% deviation on all specifications except TRP (See note 4).

FIGURE I-2
PUREX TWO-CYCLE
FLOWSHEET PPFS IV

FINAL URANIUM CYCLE
FINAL PLUTONIUM CYCLE
PLUTONIUM ION EXCHANGE

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FIGURE I-3
PUREX TWO-CYCLE
FLOWSHEET PPFS IV

WASTE CONCENTRATION
ACID RECOVERY
BACKCYCLE CONCENTRATION
ORGANIC RECOVERY

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FIGURE II

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FIGURE III

FIGURE IV

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Manager, Purex Technology
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CHEMICAL PROCESSING DEPARTMENT

Date: 11/20/58

Approved By: V. R. Cooper

Manager,
Research & Engineering
CHEMICAL PROCESSING DEPARTMENT

Date: 11/20/58

Accepted By: P. R. McMurran

Manager, Purex Operation
CHEMICAL PROCESSING DEPARTMENT

Date: 12/1/58

DECLASSIFIED

DATE

FILMED

7/6/94

END