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PROCESS BASES AND SPECIFICATIONS

THORIUM - U-233 SEPARATIONS AT THE PUREX PLANT

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By

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PROCESS BASES AND SPECIFICATIONS

THORIUM - U-233 SEPARATIONS AT THE PUREX PLANT

1. INTRODUCTION

The Purex Plant was originally designed for the chemical processing of irradiated natural uranium. It has been used nearly exclusively for this purpose during its approximately ten-year operating lifetime. However, during the winter of 1964-1965, a special processing campaign was planned and accomplished in which approximately 6 tons of irradiated thoria targets were introduced to the plant, and the thorium-232 and uranium-233 were successfully separated and purified on a demonstration basis. More recently, the planning and facilities revisions necessary for the processing of approximately 150 tons of irradiated thoria have been underway. An engineering study has been published which defines the bases for scope design of the facilities required to accomplish this operation in an acceptably safe manner(1).

For the demonstration thorium processing operation (6-ton test) of the winter of 1964-1965, process specifications were issued(2). These specifications were necessarily specific to the particular campaign inasmuch as a rather unusual processing scheme was required, by virtue of the small tonnage involved and the equipment limitations of the plant. Thus, for the relatively large operation subsequently planned, other

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(1) J. B. Kendall, "Engineering Study - Thorium Processing - Purex," RL-SEP-267 PT 1, 2, 3 & 4, May 12, 1965 (Secret).

(2) D. F. Davis, J. B. Kendall, G. A. Nicholson & S. M. Nielson, "Process Specifications for the Thorium Test in the Purex Plant," RL-SEP-36, October 30, 1964 (Secret).

[redacted]

process specifications are required. The purpose of this present document is to provide these specifications. Depending on the manner and extent of thorium - uranium-233 production developments, these present specifications may have future application, at least in part.

In addition to the process specifications, this document includes a section describing the flowsheet, and a section in which the technological bases for good process control are presented. In conjunction with the specifications, these sections are intended to provide the bases for the processing operations required to accomplish the processing objectives in a safe manner, and with minimum effect on equipment service life. All sections are organized in a manner to provide for relatively simple additions or revisions. In general, for the specifications section, appropriate levels of technical review and management approval have been required; in these cases, deviations require the same level of review and approval.

SECTION 2

FLOWSHEET

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2. FLOWSHEET

2.0 Introduction

The chemical flowsheet recommended for thorium - uranium-233 separations in the Purex Plant is presented in this section. This flowsheet has been developed for use in conjunction with the Purex Plant facilities described by RL-SEP-267 PT4(1). It is basically the same flowsheet that was presented in RL-SEP-267 PT1. Some slight revisions have been included which will improve operability and control, principally in the area of critical mass hazards control.

As herein described, the chemical flowsheet includes the following principal steps:

(a) A two-step decladding-dissolution flowsheet is used for the preparation of the dissolved Th - U-233 solution.

(b) The dissolver solution is taken to nitric acid deficiency, using an acid-boil-off and stripping technique.

(c) Solvent-extraction of the feed is accomplished, using 30 per cent TBP in diluent to effect the separation and purification of the thorium and uranium-233 products. The thorium is processed through two cycles of solvent-extraction; the uranium-233, through three.

(d) The thorium and uranium-233 are concentrated for shipping. In the case of U-233, concentration is preceded by the sorption of residual protactinium and thorium on beds of Vycor glass and cation exchange resin.

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(1) R. D. Ehrlich and J. R. LaRiviere, "Engineering Study - Thorium Processing Purex - Part IV, Process Design," RL-SEP-267 PT4, May 26, 1965 (Unclassified).

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2.1 Aluminum Can Removal

The aluminum can removal flowsheet is shown in Figure 2.0-I. The aluminum cans are removed by dissolving the aluminum in sodium hydroxide - sodium nitrate solution in A3 or B3 dissolver. The can-removal waste solution generated is routed from the dissolvers to Tank E3 for centrifugation to remove entrained thoria. The thoria centrifuge cake is washed with sodium hydroxide solution and water. The centrifuged waste solution and cake washes are combined in Tank E5 and are then sent to non-boiling waste storage. The thoria cake is slurried from the centrifuge to Tank E1 in nitric acid - potassium fluoride - aluminum nitrate solution. The thoria is digested in Tank E1 in order to dissolve most of the thoria before being recycled to Tank D1 to be combined with dissolver thorium solution.

From a chemical aspect, the aluminum can dissolution is done in the same manner as has been used in the Hanford separations plants for over 20 years for decladding aluminum-jacketed uranium elements. However, the operating technique is modified to prevent plugging the dissolver circulators and jet-out legs with particulate thoria (see Section 3.3). The flowsheet recommended for the thoria targets uses 1.50 moles NaOH/mole Al and 1.10 moles $NaNO_3$/mole Al. This is slightly higher than the current Purex practice of 1.25 moles NaOH/mole Al and 1.00 mole $NaNO_3$/mole Al in order to improve the stability of the waste solution with respect to precipitation. This is considered necessary because the thoria can-removal waste processing operation will require a longer period of time than that normally

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spent for the disposal of coating waste from Purex uranium processing. The can-removal waste solution jetted from a dissolver vessel (A3 or B3) will carry about 2 per cent of the dissolver's thoria content (approximately 160 pounds of thoria per charge).

[redacted]

2.2 Thoria Dissolution, Acid Boil-Off, and Feed Adjustment

The flowsheet for Thoria Dissolution, Acid Boil-Off, and Feed Adjustment is shown in Figure 2.0-II. The thoria is dispersed in the 13.0 M HNO_3, 0.025 M KF, 0.10 M ANN dissolvent by means of air-lift circulators. When the thoria is about 90 per cent dissolved, the thorium solution is jetted from the thoria dissolvers (A3 and B3) to Tank D1. The dissolver is then water flushed to Tank D1 in order to remove the dissolver solution heel before the next can removal operation.

Tank D1 is used as interim storage for dissolver thorium solutions, dissolver flushes, and thorium recycle from Tank E1, prior to transfer of these solutions to the C3 Acid Boil-Off Concentrator. In the C3 Acid Boil-Off Concentrator, the excess nitric acid is removed by concentration and steam distillation, and the nitric acid in the C3 Overheads is routed to the Purex Acid Recovery system. After removal of the nitric acid, the C3 solution is diluted, cooled, and jetted to Tank E6 via Tank D5 for chemical adjustment to HAF feed specifications.

The irradiated thoria received for processing will be a finely divided sand-like material which is refractory and slow to dissolve in nitric acid. (Nitric acid is the only dissolvent suitable for the existing Purex Plant stainless steel equipment.) Potassium fluoride is added to the dissolvent to make the solution 0.025 M in fluoride ion. The fluoride increases the dissolution rate of the thoria but also increases the corrosion rate of stainless steel. Aluminum is an excellent complexant for fluoride ion and substantially inhibits the corrosion of stainless steel. The presence of aluminum, however, also decreases the thoria dissolution rate. A fluoride concentration of 0.025 M

[redacted]

and an aluminum concentration of 0.10 M provide a satisfactory compromise, maximum thoria dissolution and not excessive corrosion of stainless steel.

A small heel of can removal waste solution will be left in the dissolver. This heel will add aluminum, and it will be necessary to allow for this aluminum contribution in preparing the thoria dissolvent.

The maximum concentration of nitric acid which can be produced by the Purex Acid Recovery system is 13 M. In the flowsheet presented, it was assumed that 3000 gallons working volume of nitric acid is added to the dissolver for a maximum thoria charge of 8200 pounds. If possible, the working volume of nitric acid should be increased in order to accelerate the overall thoria dissolution rate. The C3 solution is first concentrated to a thorium concentration of 4 M, and then boiled and steam sparged at a constant volume until the C3 solution becomes 0.27 M nitric acid deficient.

[redacted]

2.3 Co-Decontamination and Partition Cycle Flowsheet

The Co-Decontamination and Partition Cycle flowsheet is shown in Figure 2.0-III. The process consists of co-extraction of thorium and uranium-233 in the HA Column (T-H2) followed by partitioning of the thorium and uranium-233 in the 1BX Column (T-J6). The uranium-233 remaining in the aqueous thorium stream (1BXT) leaving the 1BX Column (T-J6) is removed by preferential extraction in the 1BS Column (T-J7). The organic scrub stream (1BSU) from the 1BS Column (T-J7) is recycled to the HA Column (T-H2) via the HAO Tank (TK-J2). The uranium product stream (1BT) from the 1BS Column (T-J7) is fed to the 1BT Concentrator (E-J8) tower for steam stripping and volume reduction to prepare the thorium solution for feed to the Second Thorium Cycle. The uranium-233 in the organic product stream (1BU) from the 1BX Column (T-J6) is stripped into an aqueous phase in the 1C Column (T-H3), and the aqueous uranium-233 stream (1CU) is fed to the 1CU Concentrator to prepare the uranium-233 for feed to the Second Uranium Cycle.

In the solvent extraction and decontamination of thorium, using a 30 per cent tributyl phosphate solvent, a low-acid, high nitrate system is desirable for good recovery and decontamination. The use of non-volatile nitrates as salting agents, however, is expensive with respect to essential materials and waste storage costs. Satisfactory recovery and decontamination of thorium can be achieved without high essential materials and waste storage costs by using an acid deficient feed (HAF) and adding a nitric acid stream (HAX-HNO_3) as a salting agent to the last stage of the HA Extraction Column (T-H2). The success of this

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flowsheet is attributed to the slow conversion rate of ZrNb-95 and Pa-233 from an inextractable species in the acid deficient feed stream (HAF) to an extractable species under the processing conditions of the HA Column (T-H2).

Phosphate is added to the HAS scrub stream to decrease the extraction of ZrNb-95 and Pa-233. Ferrous sulfamate is also added to reduce (and thus prevent the extraction of) plutonium and chromium.

Uranium and thorium are partitioned in the 1BX Column (T-J6) by control of the HAS and 1BX acidity, the 1BX Column temperature, the aqueous-to-organic flow ratio in the 1BX Column, the per cent TBP in the Purex solvent, and the HAP thorium concentration. Satisfactory partitioning of thorium and uranium is exceedingly more difficult than the current reduction method of partitioning plutonium and uranium.

Both uranium and thorium form strong complexes with dibutyl phosphate, either of which would be troublesome in the Purex solvent extraction system. With the continuous solvent washing systems of the Purex flowsheet (see 2.7, below), the probability of unacceptable concentrations of dibutyl phosphate in the solvent streams is considered to be remote. In the case of uranium, excessive dibutyl phosphate concentration would probably be manifested as high 1CW losses. In the case of thorium, the insoluble dibutyl phosphate complex is sticky and gelatinous; excessive amounts would be expected to cling to internal surfaces of the processing equipment, thus reducing operatin efficiencies and capacities.

2.4 Second and Third Uranium Cycles

The flowsheets for the Second and Third Uranium Cycles are presented in Figure 2.0-IV. The 1UC product from the First Uranium Concentrator (E-H4) overflows to the 1UC Receiver (TK-J1), is mixed with the process condensate from the Final Uranium Concentrator (E-N6), and this is pumped to the 2AF Tank (TK-J21). The Second Uranium Cycle uses the solvent extraction columns (T-J22 and T-J23) in the J-Cell Package. The 2BU stream is routed to TK-J5 and the solvent extraction columns (T-L1 and T-L2) in L-Cell are used for the Third Uranium Cycle.

No chemical adjustment is made in the 2AF Tank (TK-J21) or 3AF Tank (TK-J5). The required nitric acid (or, alternatively, ANN*) is added directly to the 2A Column (T-J22) and 3A Column (T-L1) via the middle differential-pressure taps which are located 12 inches below the feed distributor nozzle. This is a precautionary measure for criticality control purposes.

Decontamination from residual traces of plutonium is achieved by addition of ferrous sulfamate to the 2AS stream. The flowsheets for the Second Uranium Cycle and the Third Uranium Cycle are very similar to the flowsheet for the Final Plutonium Cycle during normal uranium processing.

Because of the criticality aspects of uranium-233 (discussed in the Process Control section), the concentration in the final solvent

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*The use of low acid flowsheets should not be necessary to achieve the required decontamination performance. However, if it is desirable to do so, the Second and Third Uranium Cycle flowsheets can be modified by using either aluminum nitrate or sodium nitrate in place of the nitric acid as a salting agent.

extraction product stream (3BU) is relatively low. Also for the criticality reasons, the 3BU is routed directly to TK-N1 rather than the stripper (T-L3).

A strong complex is formed between uranium and dibutyl phosphate - a degradation product of TBP. Inasmuch as the concentration of uranium-233 is also very low, the presence of a relatively small quantity of DBP in the system could result in excessive uranium losses via the solvent waste streams. With continuous solvent treatment, however, the normal concentration of DBP is sufficiently low to be of no concern with respect to uranium losses.

From TK-N1, the 3BU stream is fed to the 3BU Concentrator (E-N6) via a column of Vycor glass for adsorption of Pa-233 and ZrNb-95 in series with a cation resin column for absorption of thorium. The product from the 3BU Concentrator (3UC) overflows to TK-N7, and from there is transferred to the U-233 Load-Out System.

[redacted]

2.5 Second Thorium Cycle

The flowsheet for the Second Thorium Cycle is shown in Figure 2.0-III. The 1TC stream from the 1BT Concentrator (E-J8) overflows to the 2DF Tank (TK-K1). The nitric acid required for adequate salting strength in the 2D Column (T-K2) is added via the 2DIS and 2DX streams. A terminal water scrub stream is used in the 2D Column to minimize the acidity of the final thorium product.

The variation in acidity in different portions of the 2D Column (low acidity in the upper scrub section, about one molar in the lower scrub section, and an acid-deficient feed) should provide both high ZrNb-95 and Pa-233 DF's and a good balance between ZrNb and Ru decontamination.

Removal of the contaminants of primary concern, plutonium and protactinium, is improved by the addition of ferrous ion (ferrous sulfamate) and phosphate (phosphoric acid) to the 2DF and 2DIS streams, respectively.

The organic thorium stream (2DU) is stripped with a dilute nitric acid stream (2EX) in the 2E Column (T-K3) and the thorium product stream (2ET) is fed to the 2ET Concentrator tower for steam stripping and volume reduction.

Some decontamination of the thorium from residual uranium (and ruthenium) is anticipated in the 2E Column, but the control required to achieve these operations may be too difficult for routine operation. Routine operation may require refluxing uranium in the 2E Column and periodically purging the uranium reflux to No. 2 Solvent System (R Cell) or the 2ET Concentrator (E-K4).

[redacted]

[redacted]

2.6 Waste Concentration and Acid Recovery Flowsheets

The Waste Concentration and Acid Recovery systems as shown in Figure 2.0-V are operated in the same manner during thorium-uranium^{233} processing as during uranium-plutonium processing, except: (a) the waste solutions (2WF) accumulated in TK-F10 are combined with the HAW waste from the HA Column (T-H2) in TK-F12; (b) the acid overheads from the Acid Boil-Off Concentrator (C3) are routed to TK-F3; (c) aluminum nitrate is added to TK-F3 to complex any fluoride in the Acid Recovery system and the fluoride added to the thoria dissolvers (A3 and B3); and (d) the Acid Recovery system is operated in a manner that will produce 60 per cent nitric acid (rather than the usual 50 per cent).

The Acid Vacuum Fractionization system is operated to produce 60 per cent rather than 50 per cent nitric acid because of the increased thoria dissolution rate and increased dissolver capacity that will result. Aluminum nitrate is added to TK-F3 in order to reduce fluoride corrosion in the acid recovery system. Depending on the volatility of fluoride from the 1WW Concentrator (E-F6) and the Acid Boil-Off Concentrator (C3), it may be desirable to add the aluminum nitrate to the E-F5 tower and/or the C3 Downdraft Condenser rather than to TK-F3.

The acid overheads from the Acid Boil-Off Concentrator (C3) are added to TK-F3 in order to maximize the capacity of the Waste Concentration system. Routings, however, have been provided for diverting the stream to TK-F10 in the event the routing to TK-F3 results in unacceptable radiation levels in the Acid Recovery system.

[redacted]

Even with the C3 overheads routed to TK-F3, the sugar denitration vessels (TK-F15 and TK-F16) must be operated at maximum capacity in order to handle the high level waste (1WW) flow. Operation of the sugar denitration system in this manner will increase nitric acid losses via the Acid Absorber (T-F5) overheads.

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2.7 Solvent Treatment Flowsheets

The No. 1 and No. 2 Solvent Treatment Systems as shown in Figure 2.0-VI are operated in the same manner during thorium-uranium^{233} processing as during uranium-plutonium processing except: (a) wash solutions from the No. 2 system are not reused in the No. 1 system; (b) potassium permanganate is not added continuously to TK-G1; and (c) the 2OS-HNO_3 flow is substantially increased. The reasons for the modifications are: (a) reuse of wash solutions from the No. 2 system is not recommended because plant experience during the thorium test run of January, 1965 indicated a solvent quality problem may be encountered during thorium processing and the study of a solvent quality problem will be substantially simplified by isolating the two systems; (b) the continuous addition of potassium permanganate to the 1OF Tank (TK-G1) is not necessary because of the low ZrNb-95 activity in the 1CW stream; and (c) thorium extraction and decontamination is improved and high level waste volumes are reduced by the addition of nitric acid to the last extraction stage of a thorium extraction column. Since a distributor is not available for adding nitric acid directly to the last extraction stage of the 2D Column (T-K2), the nitric acid is added via the 2DX stream.

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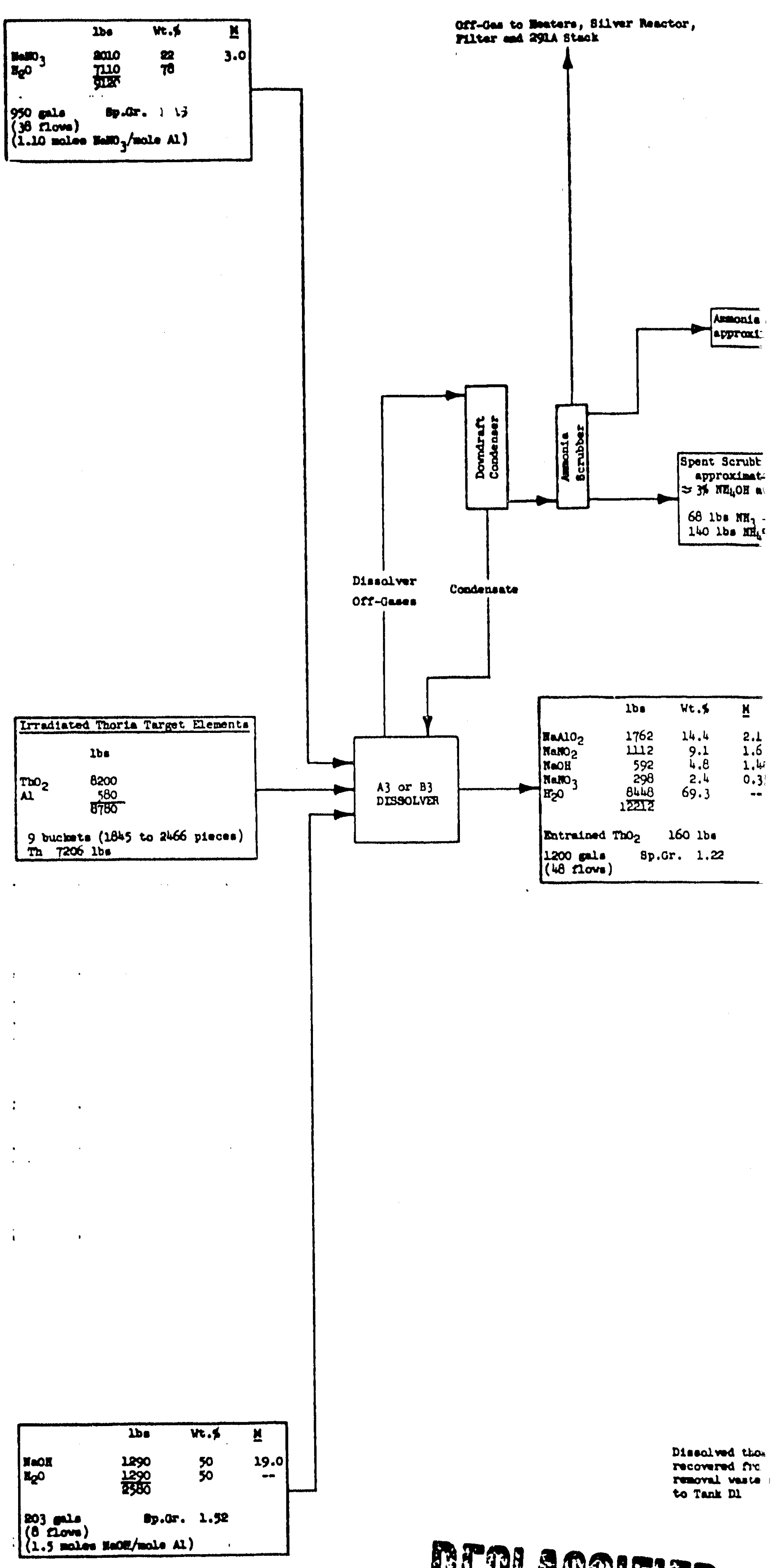

	lbs	Wt.%	M
$NaNO_3$	2010	22	3.0
H_2O	7110	78	
	9120		

950 gals Sp.Gr. 1.13
(38 flows)
(1.10 moles $NaNO_3$/mole Al)

Irradiated Thoria Target Elements

	lbs
ThO_2	8200
Al	580
	8780

9 buckets (1845 to 2466 pieces)
Th 7206 lbs

	lbs	Wt.%	M
$NaAlO_2$	1762	14.4	2.1
$NaNO_2$	1112	9.1	1.6
$NaOH$	592	4.8	1.4
$NaNO_3$	298	2.4	0.3
H_2O	8448	69.3	--
	12212		

Entrained ThO_2 160 lbs
1200 gals Sp.Gr. 1.22
(48 flows)

	lbs	Wt.%	M
$NaOH$	1290	50	19.0
H_2O	1290	50	--
	2580		

203 gals Sp.Gr. 1.52
(8 flows)
(1.5 moles NaOH/mole Al)

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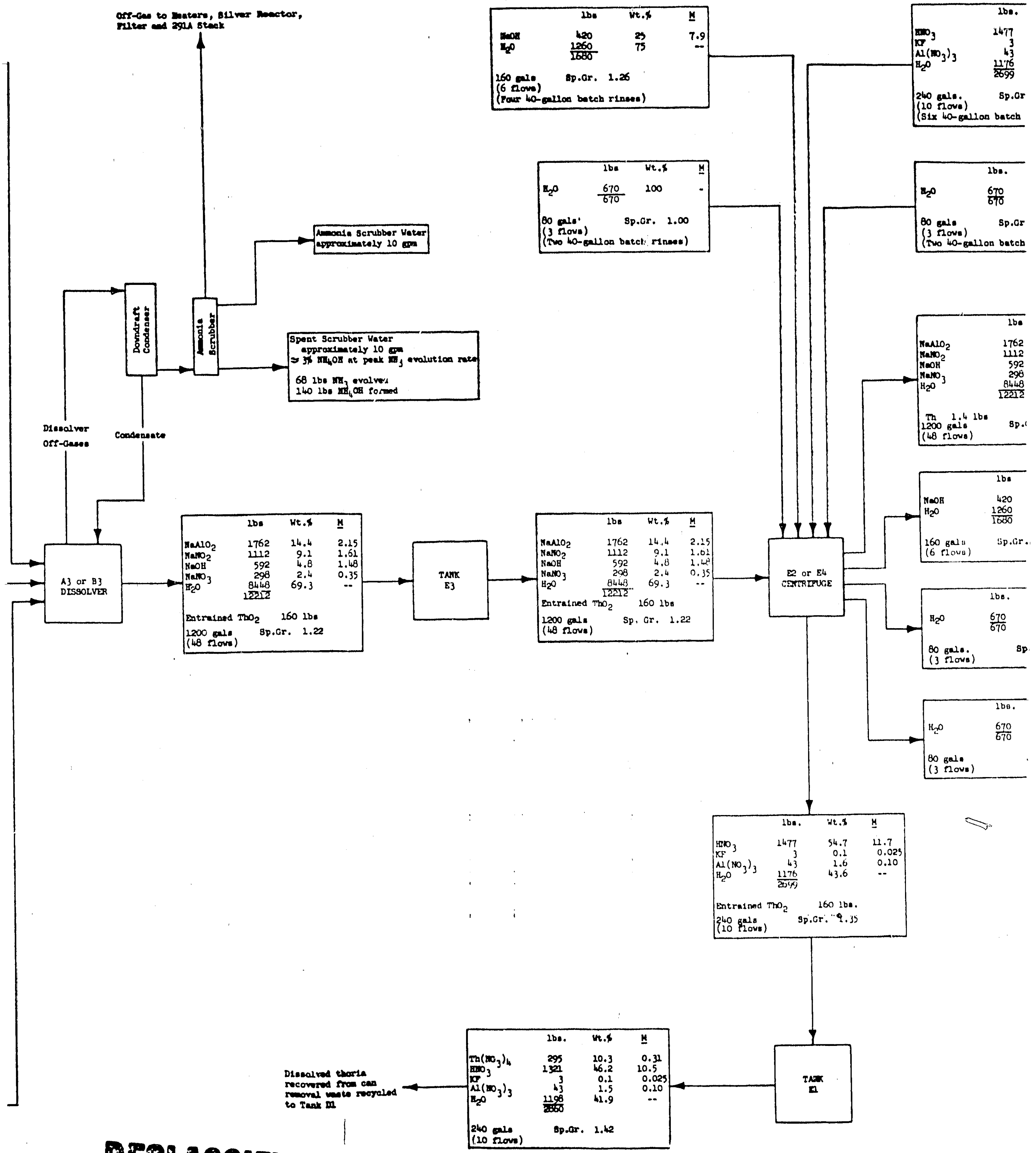

Off-Gas to Heaters, Silver Reactor, Filter and 291A Stack
Ammonia Scrubber Water approximately 10 gpm
Downdraft Condenser
Ammonia Scrubber
Spent Scrubber Water
approximately 10 gpm
≈ 3% NH_4OH at peak NH_3 evolution rate
68 lbs NH_3 evolved
140 lbs NH_4OH formed
Dissolver Off-Gases
Condensate
A3 or B3 DISSOLVER
lbs Wt.% M
$NaAlO_2$ 1762 14.4 2.15
$NaNO_2$ 1112 9.1 1.61
NaOH 592 4.8 1.48
$NaNO_3$ 298 2.4 0.35
H_2O 8448 69.3 --
12212
Entrained ThO_2 160 lbs
1200 gals Sp.Gr. 1.22
(48 flows)
TANK E3
lbs Wt.% M
$NaAlO_2$ 1762 14.4 2.15
$NaNO_2$ 1112 9.1 1.61
NaOH 592 4.8 1.48
$NaNO_3$ 298 2.4 0.35
H_2O 8448 69.3 --
12212
Entrained ThO_2 160 lbs
1200 gals Sp. Gr. 1.22
(48 flows)
lbs Wt.% M
NaOH 420 25 7.9
H_2O 1260 75 --
1680
160 gals Sp.Gr. 1.26
(6 flows)
(Four 40-gallon batch rinses)
lbs Wt.% M
H_2O 670 100 -
670
80 gals Sp.Gr. 1.00
(3 flows)
(Two 40-gallon batch rinses)
lbs.
HNO_3 1477
KF 3
$Al(NO_3)_3$ 43
H_2O 1176
2699
240 gals. Sp.Gr
(10 flows)
(Six 40-gallon batch
lbs.
H_2O 670
670
80 gals Sp.Gr
(3 flows)
(Two 40-gallon batch
E2 or E4 CENTRIFUGE
lbs
$NaAlO_2$ 1762
$NaNO_2$ 1112
NaOH 592
$NaNO_3$ 298
H_2O 8448
12212
Th 1.4 lbs
1200 gals Sp.
(48 flows)
lbs
NaOH 420
H_2O 1260
1680
160 gals Sp.Gr.
(6 flows)
lbs.
H_2O 670
670
80 gals. Sp
(3 flows)
lbs.
H_2O 670
670
80 gals
(3 flows)
lbs. Wt.% M
HNO_3 1477 54.7 11.7
KF 3 0.1 0.025
$Al(NO_3)_3$ 43 1.6 0.10
H_2O 1176 43.6 --
2699
Entrained ThO_2 160 lbs.
240 gals Sp.Gr. 1.35
(10 flows)
TANK E1
lbs. Wt.% M
$Th(NO_3)_4$ 295 10.3 0.31
HNO_3 1321 46.2 10.5
KF 3 0.1 0.025
$Al(NO_3)_3$ 43 1.5 0.10
H_2O 1198 41.9 --
2860
240 gals Sp.Gr. 1.42
(10 flows)
Dissolved thoria recovered from can removal waste recycled to Tank E1

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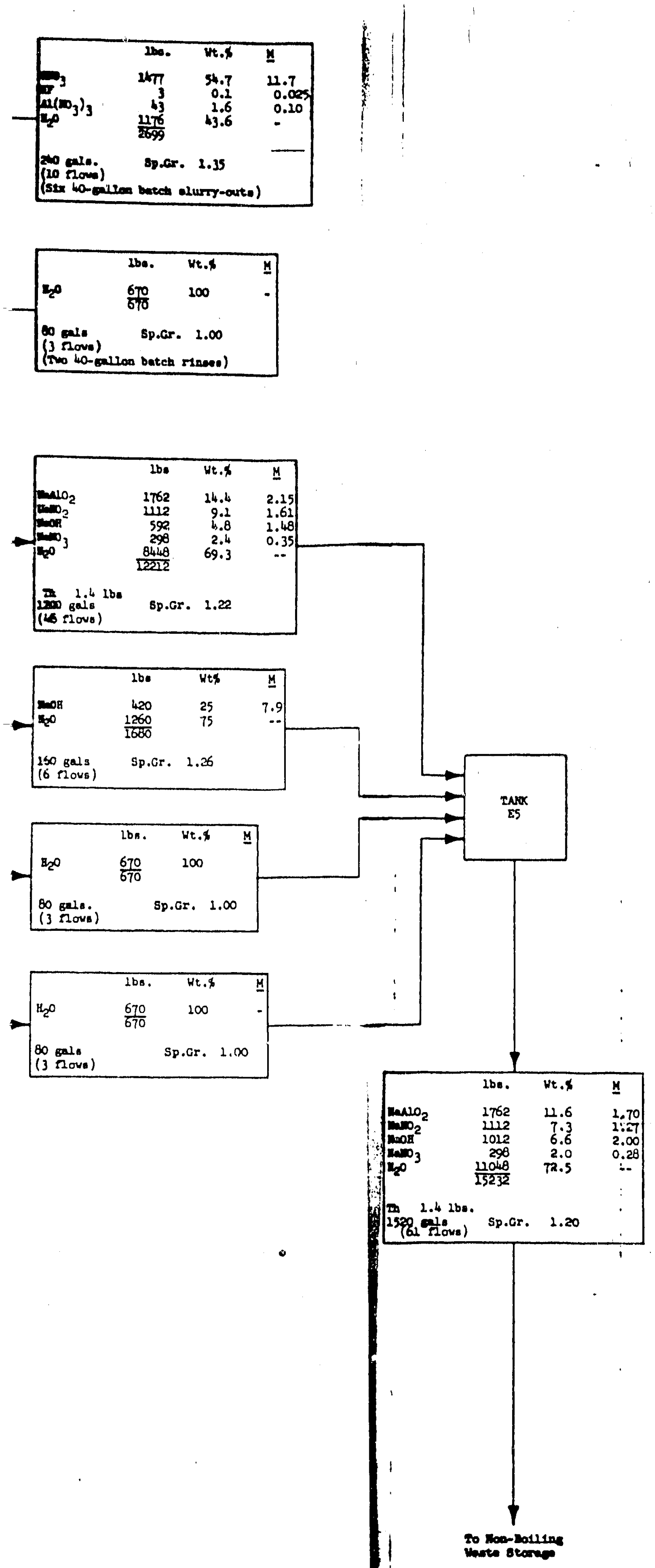

	lbs.	Wt.%	M
HNO_3	1477	54.7	11.7
HF	3	0.1	0.025
$Al(NO_3)_3$	43	1.6	0.10
H_2O	1176	43.6	-
	2699		

240 gals. Sp.Gr. 1.35
(10 flows)
(Six 40-gallon batch slurry-outs)

	lbs.	Wt.%	M
H_2O	670	100	-
	670		

80 gals Sp.Gr. 1.00
(3 flows)
(Two 40-gallon batch rinses)

	lbs	Wt.%	M
$NaAlO_2$	1762	14.4	2.15
$NaNO_2$	1112	9.1	1.61
$NaOH$	592	4.8	1.48
$NaNO_3$	298	2.4	0.35
H_2O	8448	69.3	--
	12212		

Th 1.4 lbs
1200 gals Sp.Gr. 1.22
(46 flows)

	lbs	Wt%	M
$NaOH$	420	25	7.9
H_2O	1260	75	--
	1680		

150 gals Sp.Gr. 1.26
(6 flows)

	lbs.	Wt.%	M
H_2O	670	100	
	670		

80 gals. Sp.Gr. 1.00
(3 flows)

	lbs.	Wt.%	M
H_2O	670	100	-
	670		

80 gals Sp.Gr. 1.00
(3 flows)

	lbs.	Wt.%	M
$NaAlO_2$	1762	11.6	1.70
$NaNO_2$	1112	7.3	1.27
$NaOH$	1012	6.6	2.00
$NaNO_3$	298	2.0	0.28
H_2O	11048	72.5	--
	15232		

Th 1.4 lbs.
1520 gals Sp.Gr. 1.20
(61 flows)

FIGURE 2.0-I

PURFX THORIUM PROCESS FLOWSHEET
ALUMINUM CAN REMOVAL

Basis: 1 Dissolver Charge
8200 lbs. ThO_2
580 lbs. Al

100 flows = 690 gallons per ton thorium in the HAF

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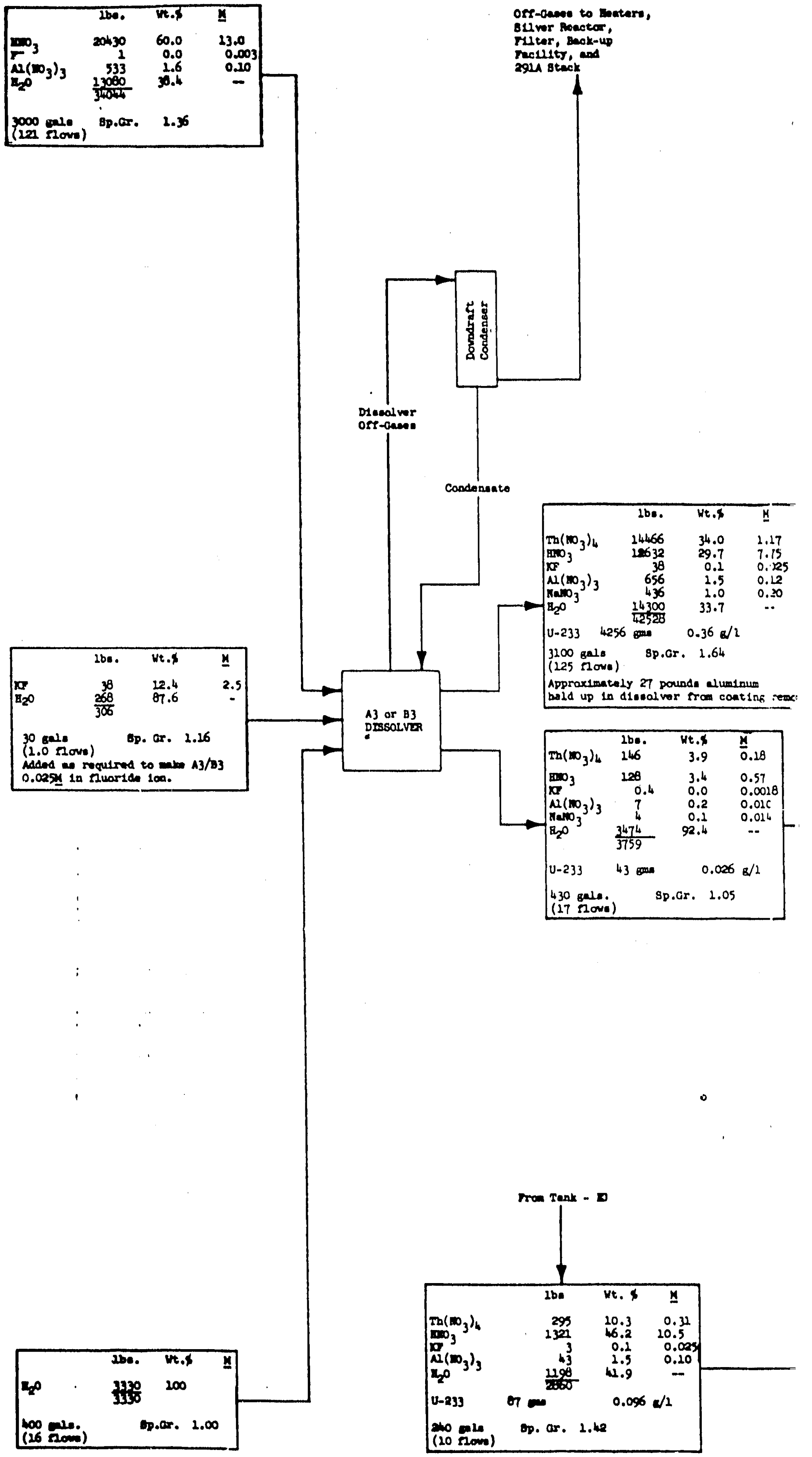


	lbs.	Wt.%	M
HNO_3	20430	60.0	13.0
F^-	1	0.0	0.003
$Al(NO_3)_3$	533	1.6	0.10
H_2O	13080	38.4	--
	34044		

3000 gals Sp.Gr. 1.36
(121 flows)

	lbs.	Wt.%	M
KF	38	12.4	2.5
H_2O	268	87.6	-
	306		

30 gals Sp. Gr. 1.16
(1.0 flows)
Added as required to make A3/B3 0.025M in fluoride ion.

	lbs.	Wt.%	M
H_2O	3330	100	
	3330		

400 gals. Sp.Gr. 1.00
(16 flows)

	lbs.	Wt.%	M
$Th(NO_3)_4$	14466	34.0	1.17
HNO_3	12632	29.7	7.75
KF	38	0.1	0.025
$Al(NO_3)_3$	656	1.5	0.12
$NaNO_3$	436	1.0	0.20
H_2O	14300	33.7	--
	42528		

U-233 4256 gms 0.36 g/l

3100 gals Sp.Gr. 1.64
(125 flows)
Approximately 27 pounds aluminum held up in dissolver from coating rem

	lbs.	Wt.%	M
$Th(NO_3)_4$	146	3.9	0.18
HNO_3	128	3.4	0.57
KF	0.4	0.0	0.0018
$Al(NO_3)_3$	7	0.2	0.010
$NaNO_3$	4	0.1	0.014
H_2O	3474	92.4	--
	3759		

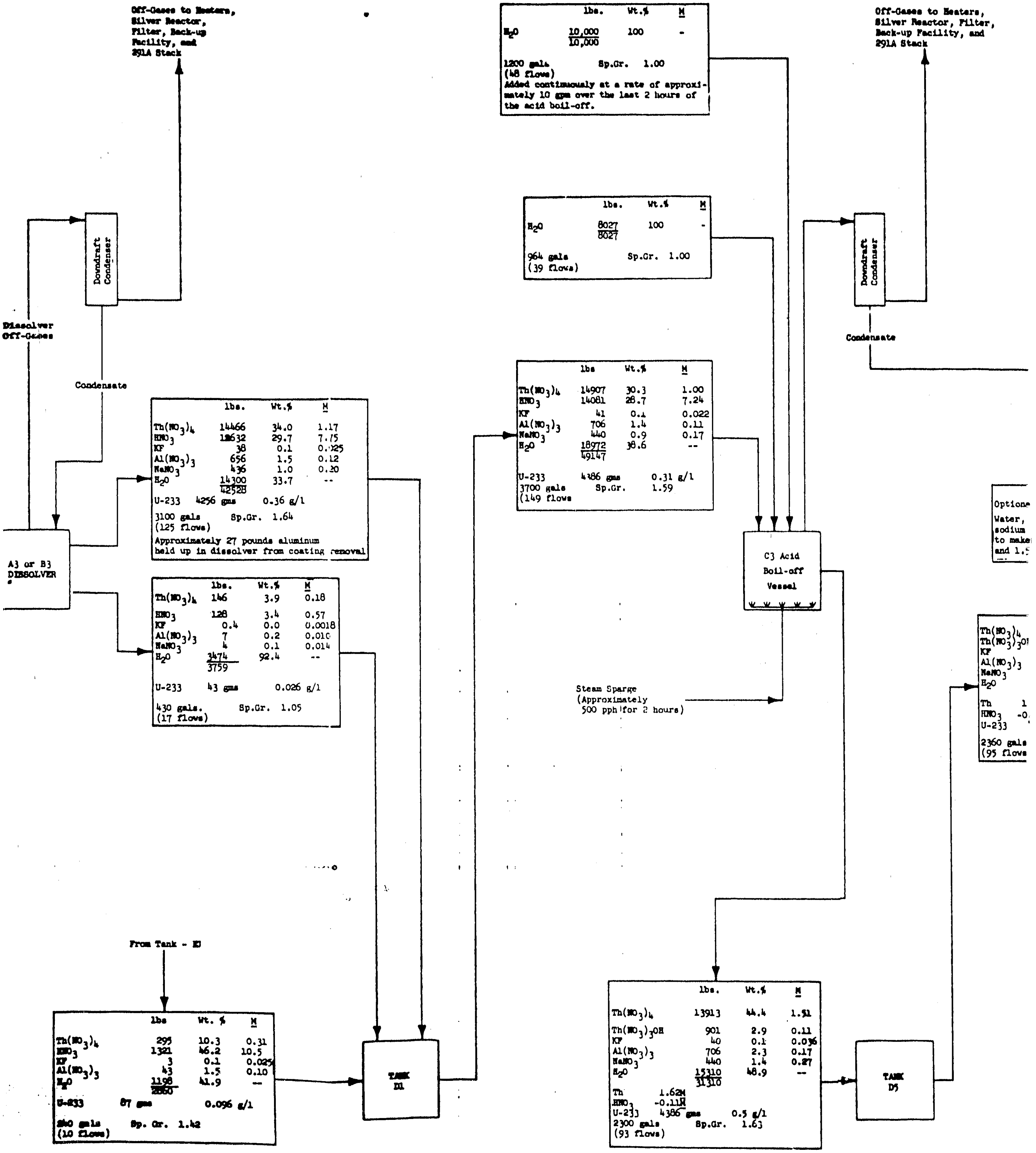
U-233 43 gms 0.026 g/l

430 gals. Sp.Gr. 1.05
(17 flows)

	lbs	Wt. %	M
$Th(NO_3)_4$	295	10.3	0.31
HNO_3	1321	46.2	10.5
KF	3	0.1	0.025
$Al(NO_3)_3$	43	1.5	0.10
H_2O	1198	41.9	--
	2860		

U-233 87 gms 0.096 g/l

240 gals Sp. Gr. 1.42
(10 flows)



	lbs.	Wt.%	M
H_2O	10,000	100	-
	10,000		

1200 gals. Sp.Gr. 1.00
(48 flows)
Added continuously at a rate of approximately 10 gpm over the last 2 hours of the acid boil-off.

	lbs.	Wt.%	M
H_2O	8027	100	-
	8027		

964 gals Sp.Gr. 1.00
(39 flows)

	lbs.	Wt.%	M
$Th(NO_3)_4$	14466	34.0	1.17
HNO_3	12632	29.7	7.75
KF	38	0.1	0.025
$Al(NO_3)_3$	656	1.5	0.12
$NaNO_3$	436	1.0	0.20
H_2O	14300	33.7	--
	42528		

U-233 4256 gms 0.36 g/l
3100 gals Sp.Gr. 1.64
(125 flows)
Approximately 27 pounds aluminum held up in dissolver from coating removal

	lbs.	Wt.%	M
$Th(NO_3)_4$	146	3.9	0.18
HNO_3	128	3.4	0.57
KF	0.4	0.0	0.0018
$Al(NO_3)_3$	7	0.2	0.010
$NaNO_3$	4	0.1	0.014
H_2O	3474	92.4	--
	3759		

U-233 43 gms 0.026 g/l
430 gals. Sp.Gr. 1.05
(17 flows)

	lbs	Wt.%	M
$Th(NO_3)_4$	14907	30.3	1.00
HNO_3	14081	28.7	7.24
KF	41	0.1	0.022
$Al(NO_3)_3$	706	1.4	0.11
$NaNO_3$	440	0.9	0.17
H_2O	18972	38.6	--
	49147		

U-233 4386 gms 0.31 g/l
3700 gals Sp.Gr. 1.59
(149 flows

	lbs	Wt. %	M
$Th(NO_3)_4$	295	10.3	0.31
HNO_3	1321	46.2	10.5
KF	3	0.1	0.025
$Al(NO_3)_3$	43	1.5	0.10
H_2O	1198	41.9	--
	2860		

U-233 87 gms 0.096 g/l
240 gals Sp. Gr. 1.42
(10 flows)

	lbs.	Wt.%	M
$Th(NO_3)_4$	13913	44.4	1.51
$Th(NO_3)_3OH$	901	2.9	0.11
KF	40	0.1	0.036
$Al(NO_3)_3$	706	2.3	0.17
$NaNO_3$	440	1.4	0.27
H_2O	15310	48.9	--
	31310		

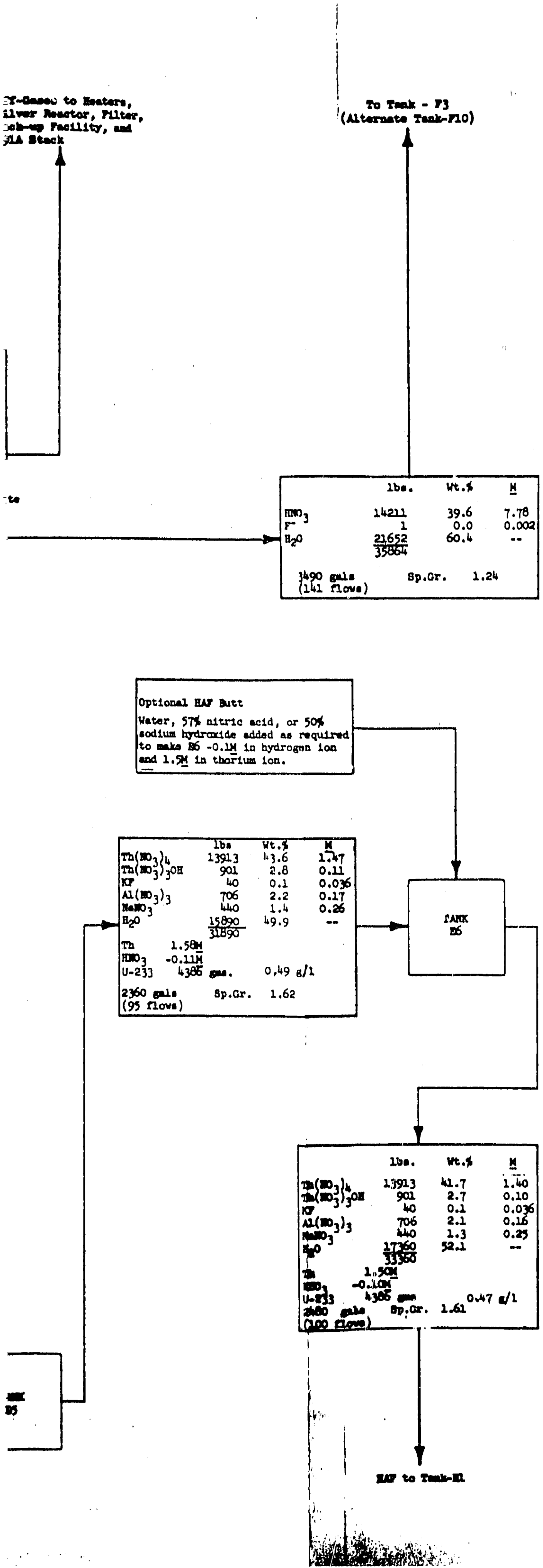
Th 1.62M
HNO_3 -0.11M
U-233 4386 gms 0.5 g/l
2300 gals Sp.Gr. 1.63
(93 flows)

Optione
Water,
sodium
to make
and 1.5

$Th(NO_3)_4$
$Th(NO_3)_3O$
KF
$Al(NO_3)_3$
$NaNO_3$
H_2O

Th 1
HNO_3 -0,
U-233

2360 gals
(95 flows



	lbs.	Wt.%	M
HNO_3	14211	39.6	7.78
F^-	1	0.0	0.002
H_2O	21652	60.4	--
	35864		

3490 gals Sp.Gr. 1.24
(141 flows)

Optional HAF Butt
Water, 57% nitric acid, or 50% sodium hydroxide added as required to make E6 -0.1M in hydrogen ion and 1.5M in thorium ion.

	lbs	Wt.%	M
$Th(NO_3)_4$	13913	43.6	1.47
$Th(NO_3)_3OH$	901	2.8	0.11
KF	40	0.1	0.036
$Al(NO_3)_3$	706	2.2	0.17
$NaNO_3$	440	1.4	0.26
H_2O	15890	49.9	--
	31890		

Th 1.58M
HNO_3 -0.11M
U-233 4386 gms. 0,49 g/l
2360 gals Sp.Gr. 1.62
(95 flows)

	lbs.	Wt.%	M
$Th(NO_3)_4$	13913	41.7	1.40
$Th(NO_3)_3OH$	901	2.7	0.10
KF	40	0.1	0.036
$Al(NO_3)_3$	706	2.1	0.16
$NaNO_3$	440	1.3	0.25
H_2O	17360	52.1	--
	33360		

Th 1.50M
HNO_3 -0.10M
U-233 4386 gms 0.47 g/l
2460 gals Sp.Gr. 1.61
(100 flows)

FIGURE 2.0-II

PURSEX THORIUM PROCESS FLOWSHEET
THORIA DISSOLUTION AND
FEED ADJUSTMENT

basis: 1 Dissolver Charge
8200 lbs ThO_2

100 flows = 690 gallons per ton thorium in the HAF

DRAWN 7-15-65 D E ELIASON

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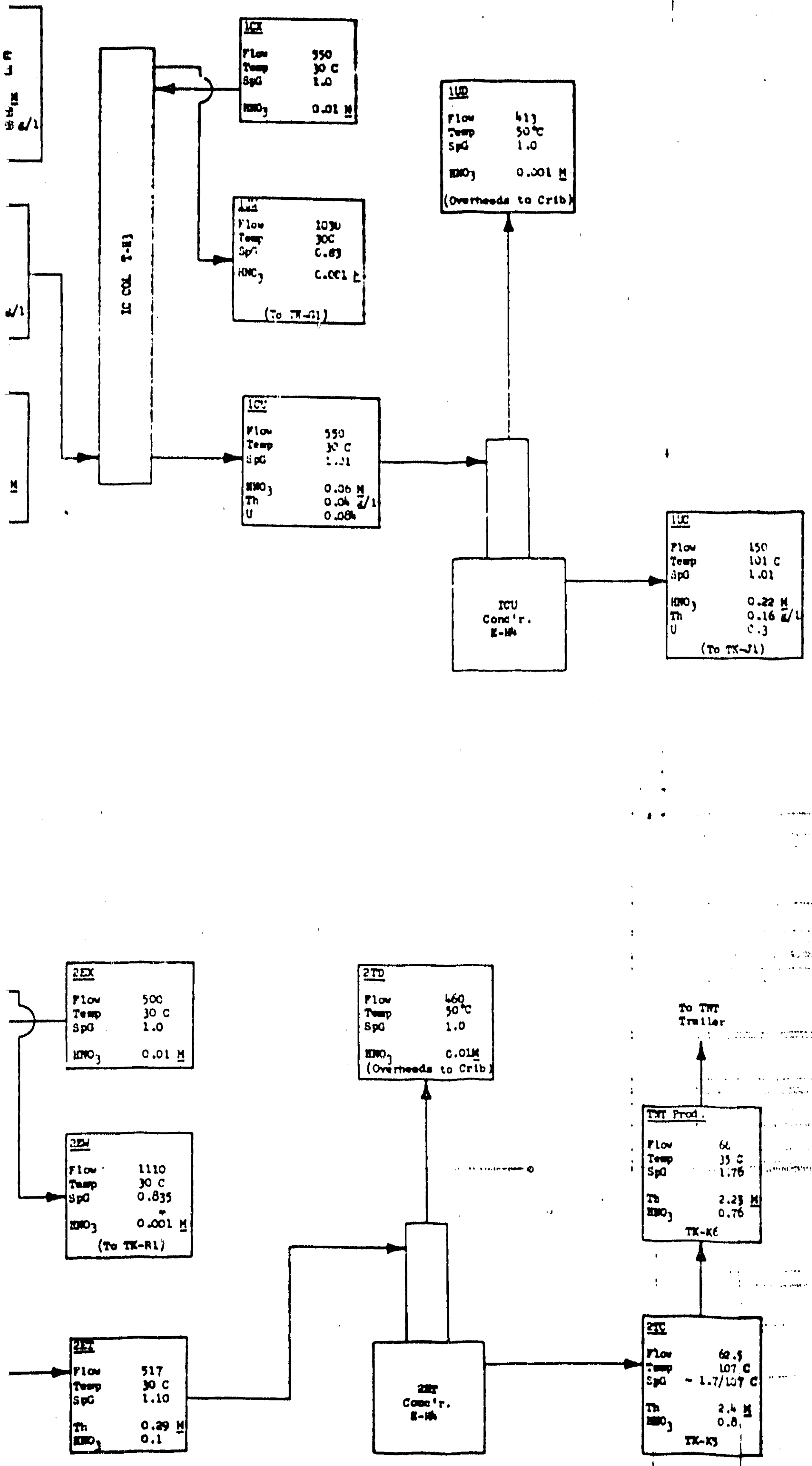
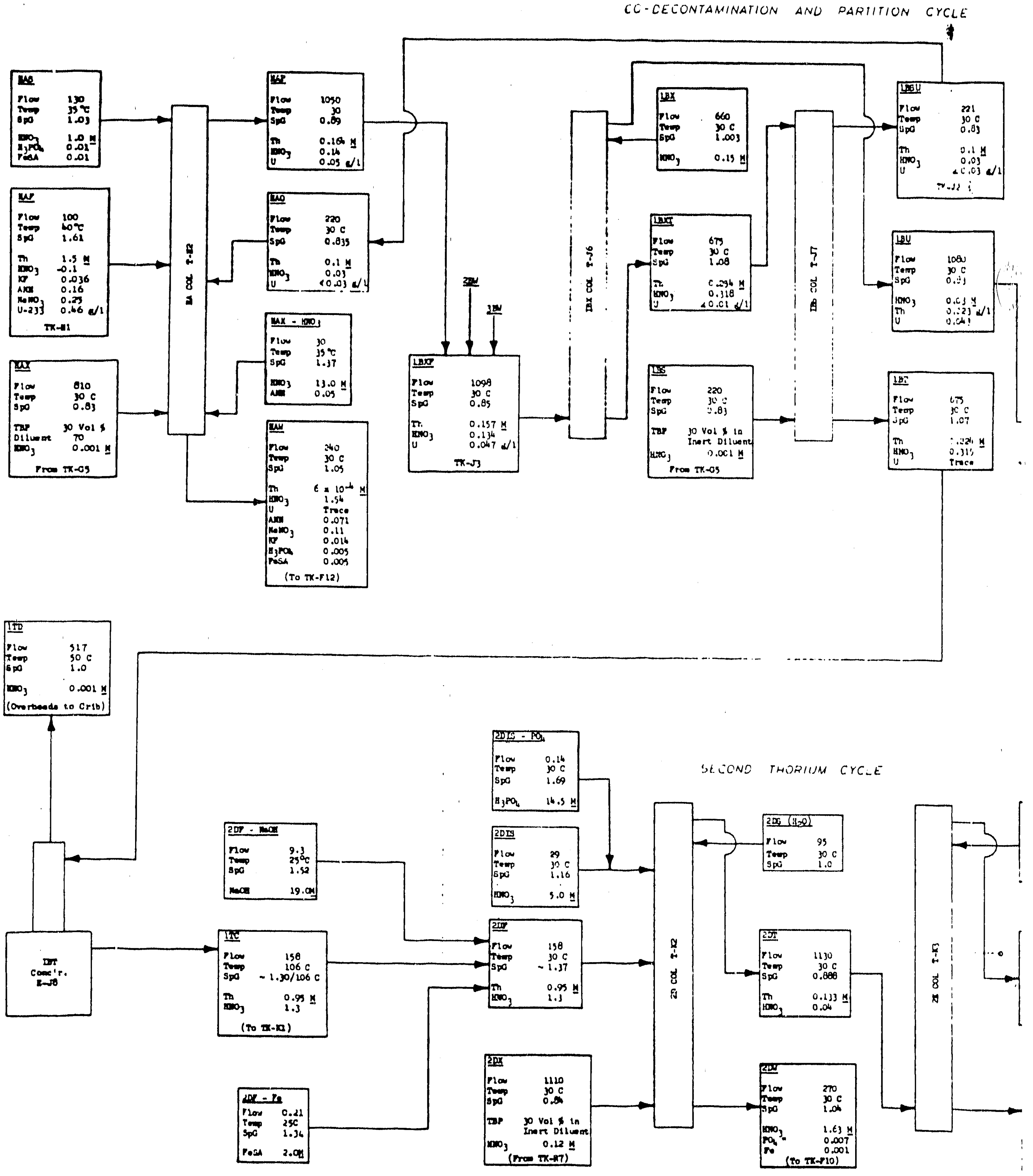


FIGURE 2.0-III

PUREX THORIUM PROCESS FLOWSHEET
CO-DECONTAMINATION AND PARTITION CYCLE
SECOND THORIUM CYCLE
DRAWN 7-15-65 D E ELIASON
[redacted] REVISED 2-23-66



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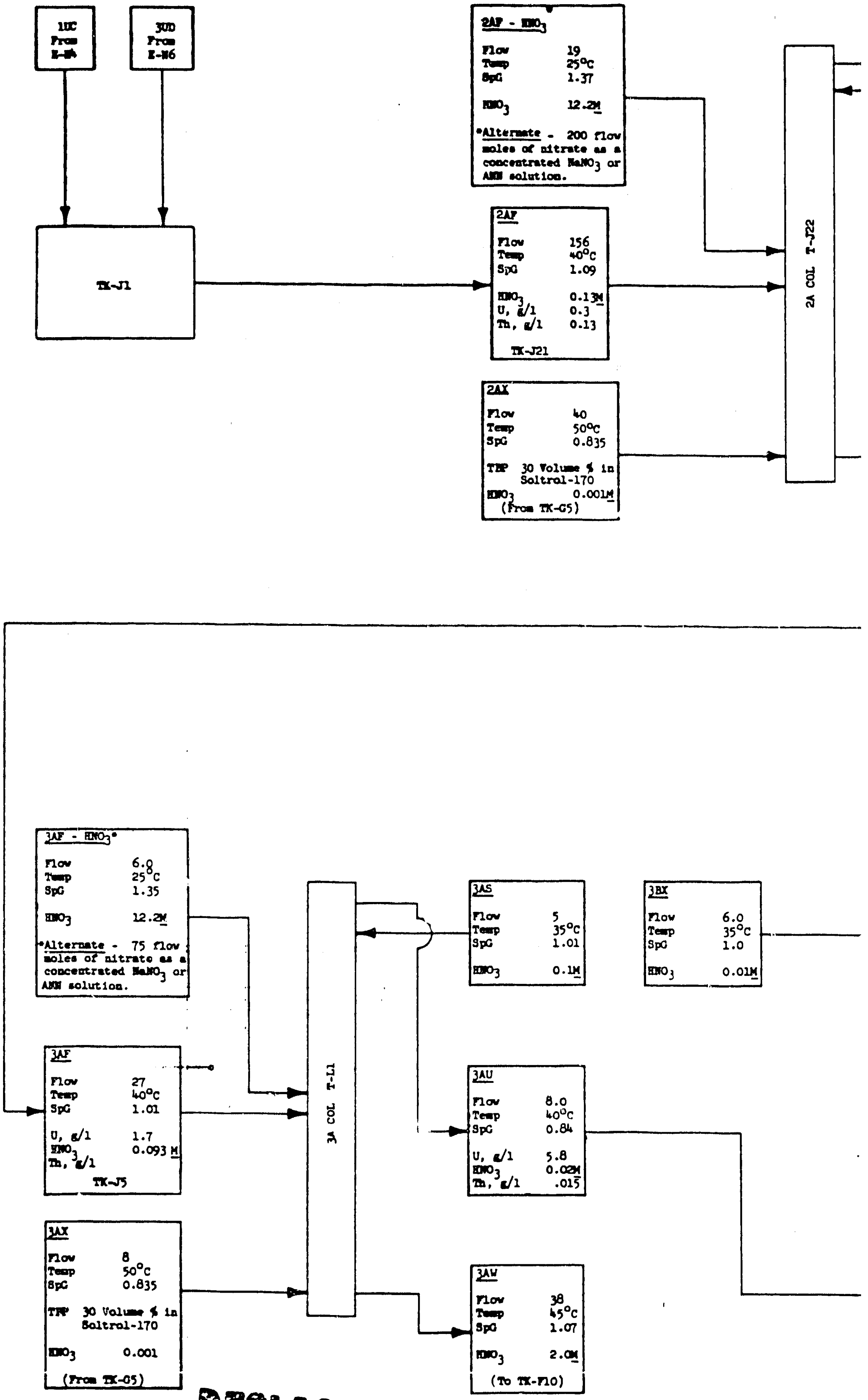

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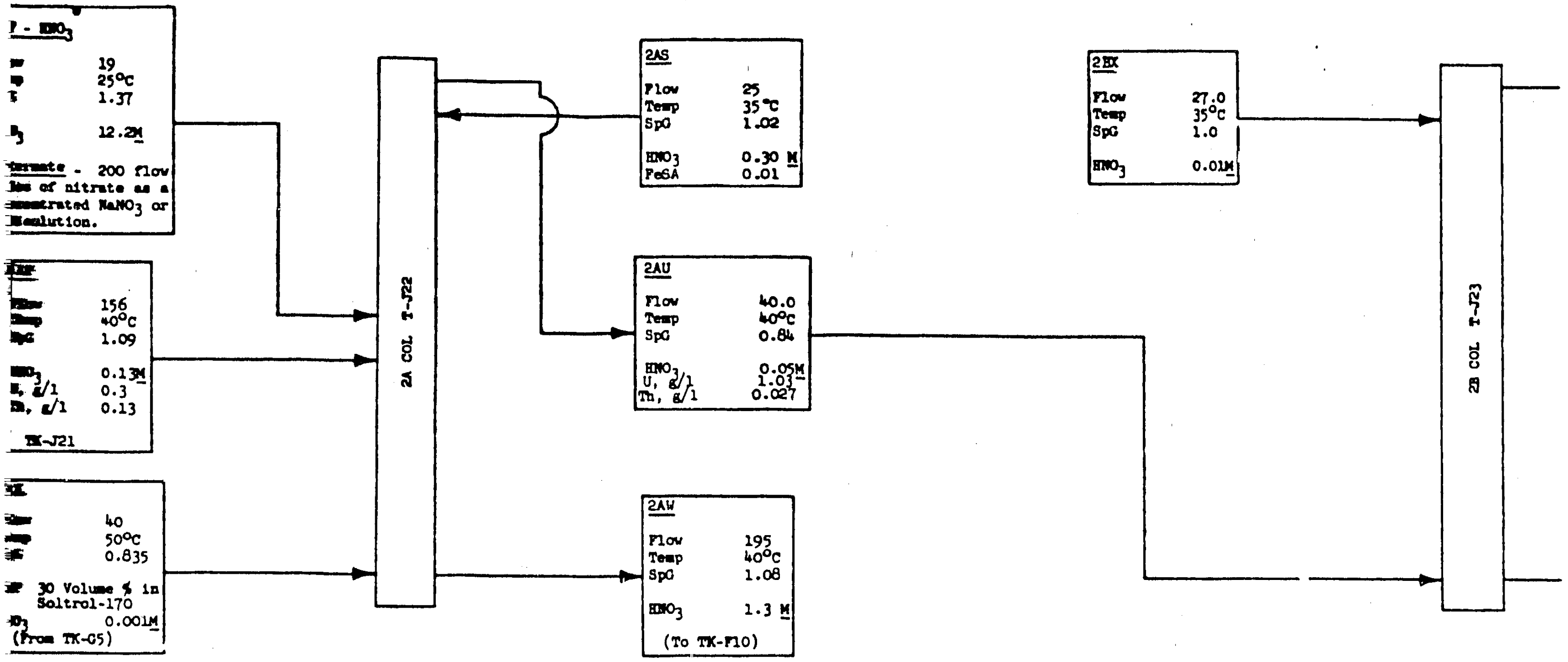
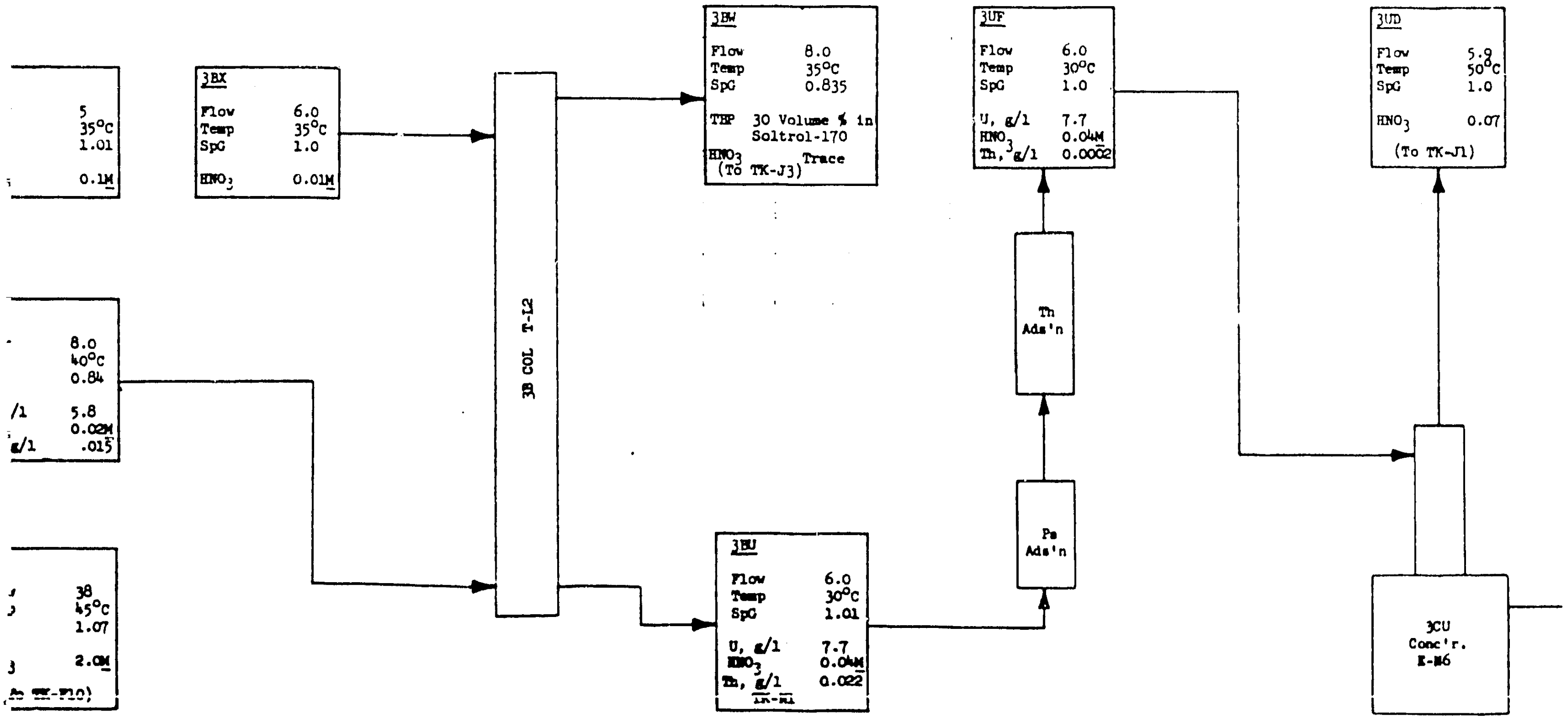
SECOND URANIUM CYCLE



THIRD URANIUM CYCLE



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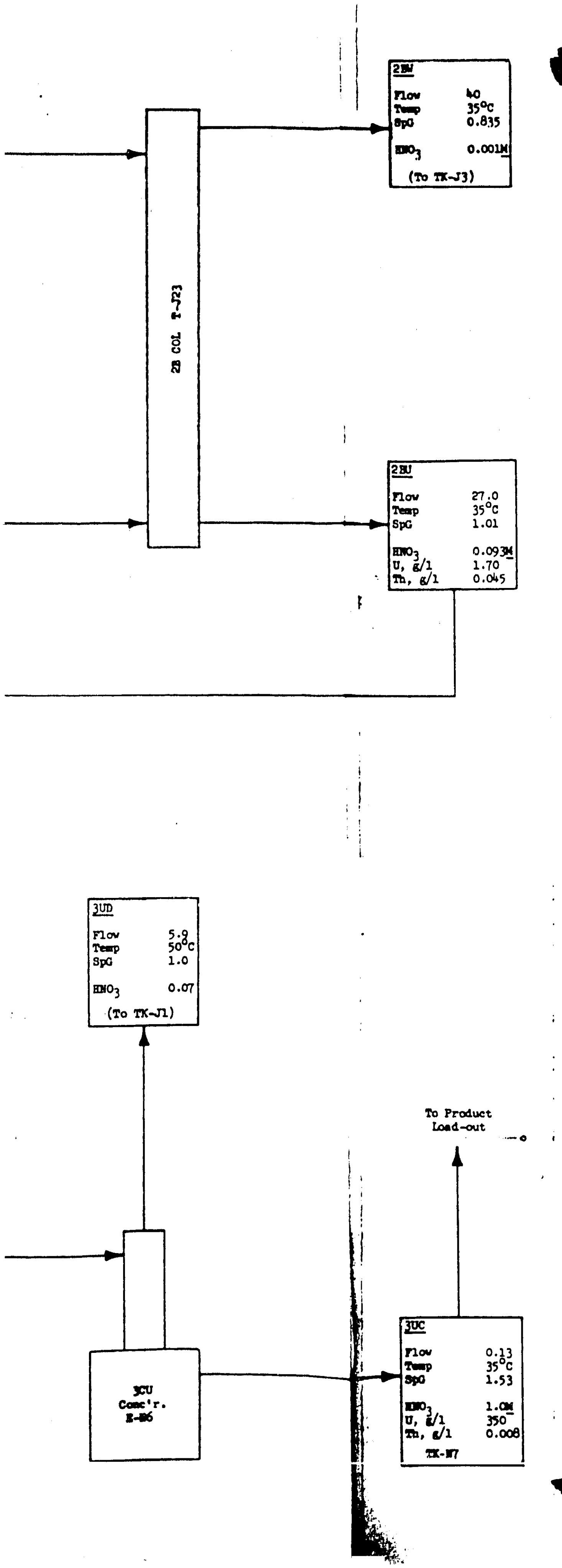


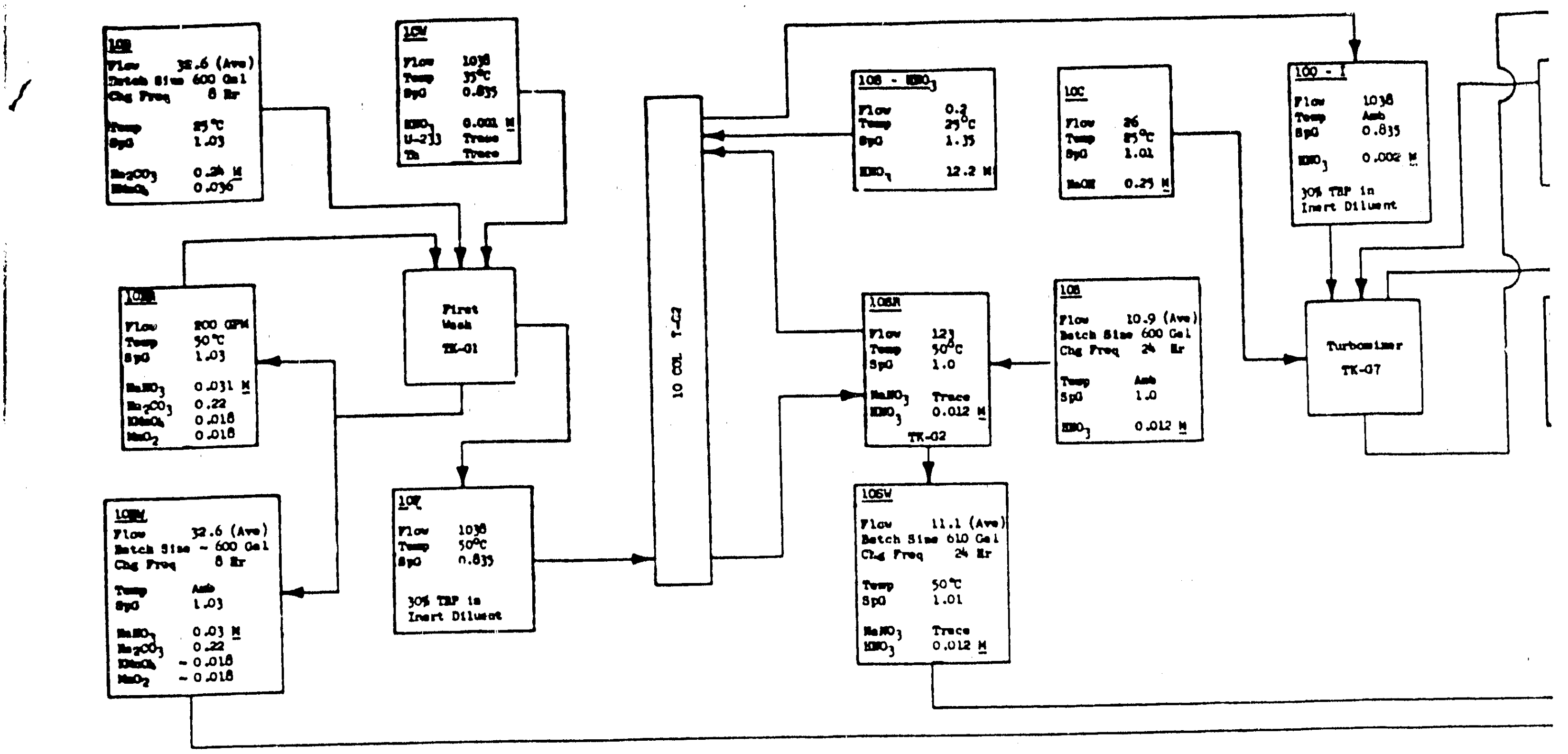
FIGURE 2.0-IV

PURAX THORIUM PROCESS FLOWSHEET
SECOND URANIUM CYCLE
THIRD URANIUM CYCLE

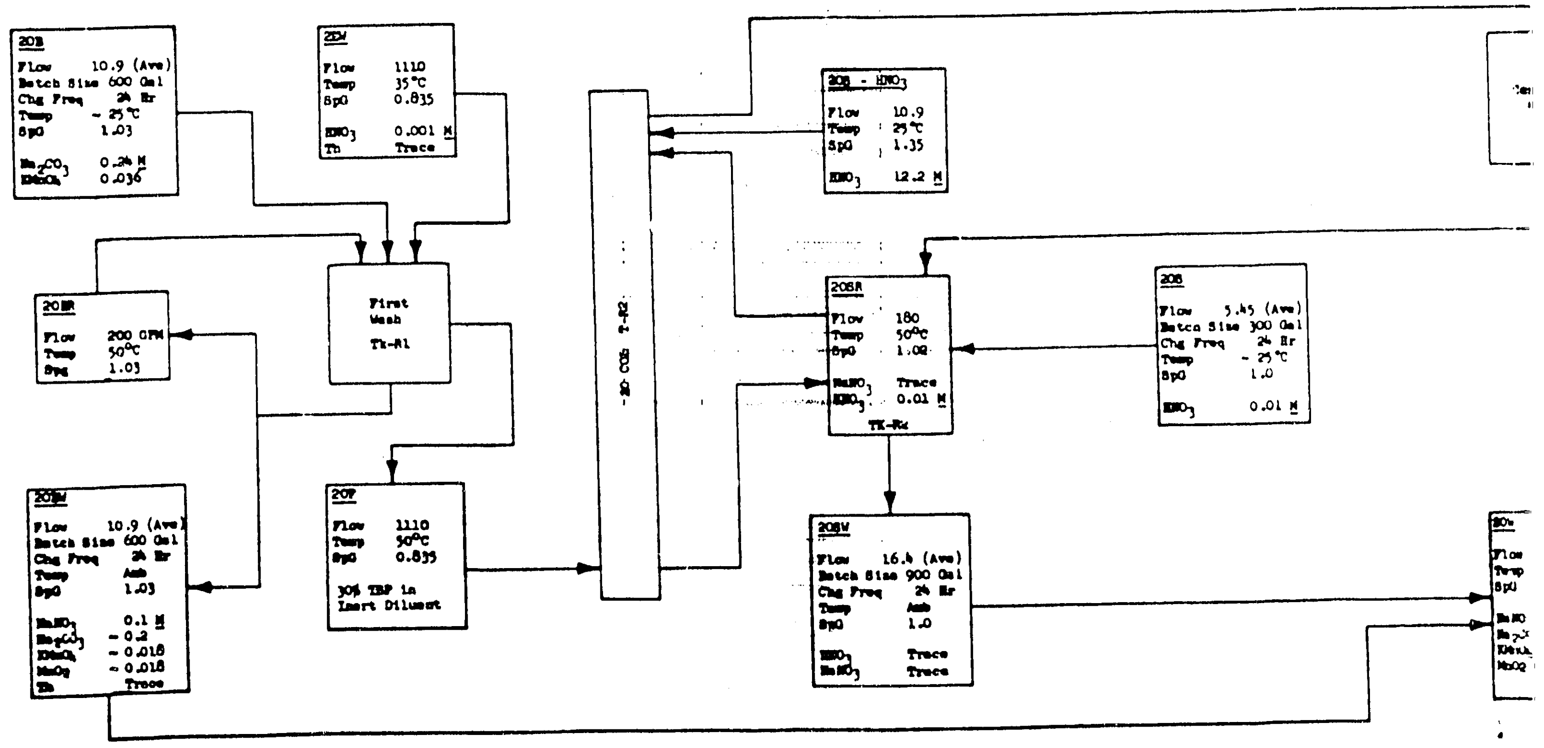
DRAWN 7-15-65 D E ELIASON

[redacted]

NO. 1 SOLVENT TREATMENT SYSTEM



NO. 2 SOLVENT TREATMENT SYSTEM



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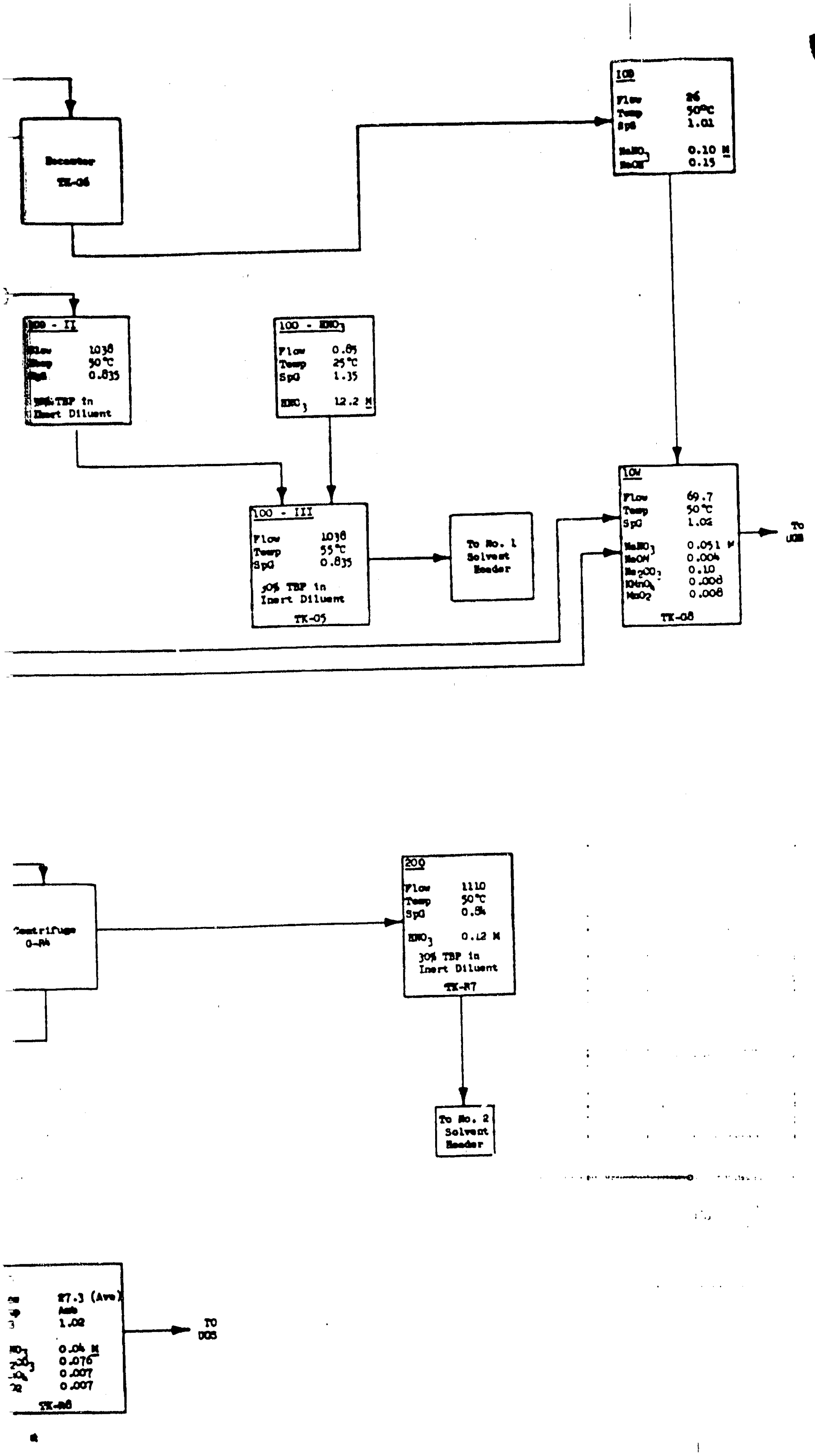

FIGURE 2.0-V

PUREX THORIUM PROCESS FLOWSHEET
NO. 1 SOLVENT TREATMENT SYSTEM
NO. 2 SOLVENT TREATMENT SYSTEM
DRAWN 7-15-65 D E ELIASON

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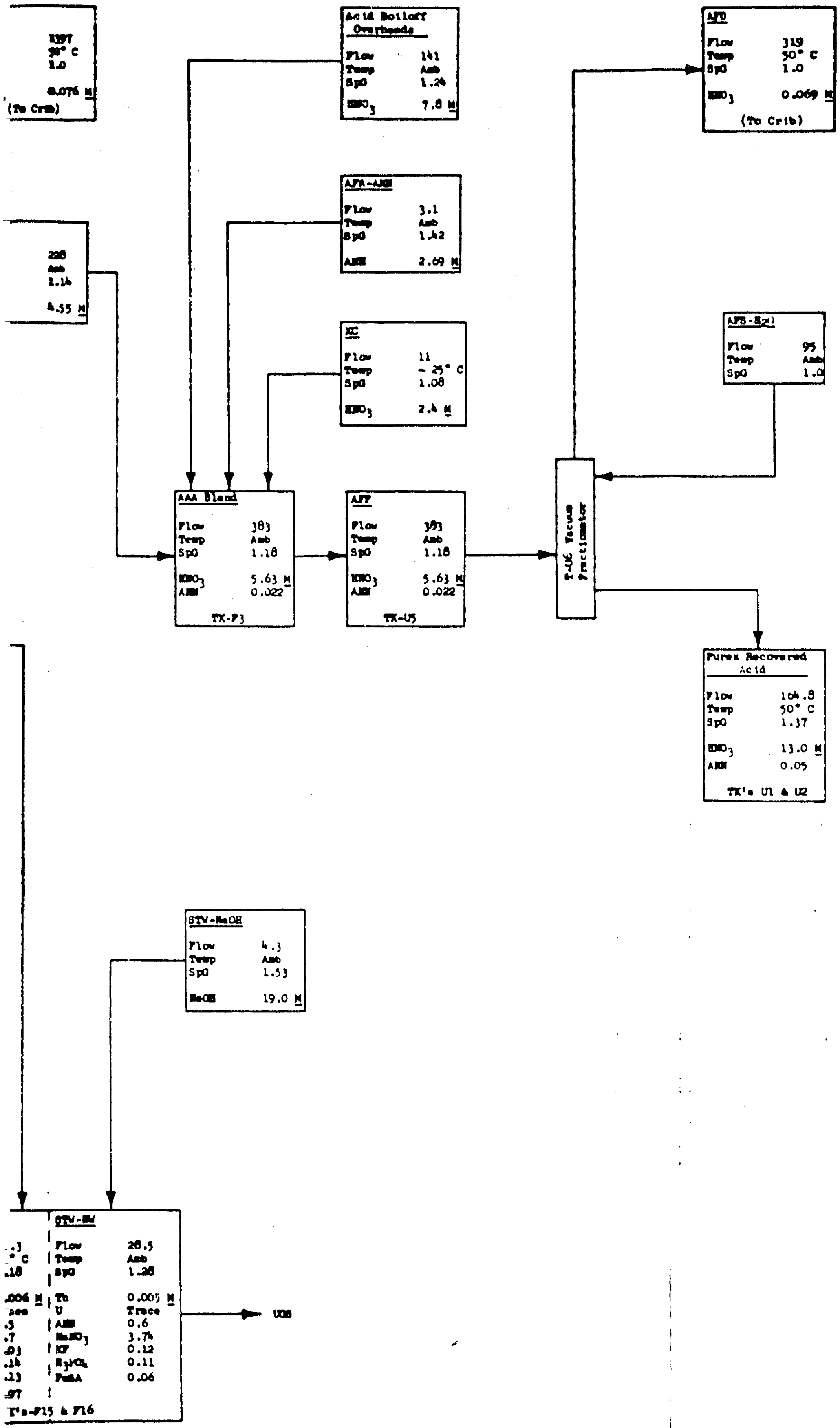

FIGURE 2.0-VI

PUREX THORIUM PROCESS FLOWSHEET
WASTE CONCENTRATION AND
ACID RECOVERY

DRAWN 8-5-65 D E ELIASON
REVISED 2-23-66

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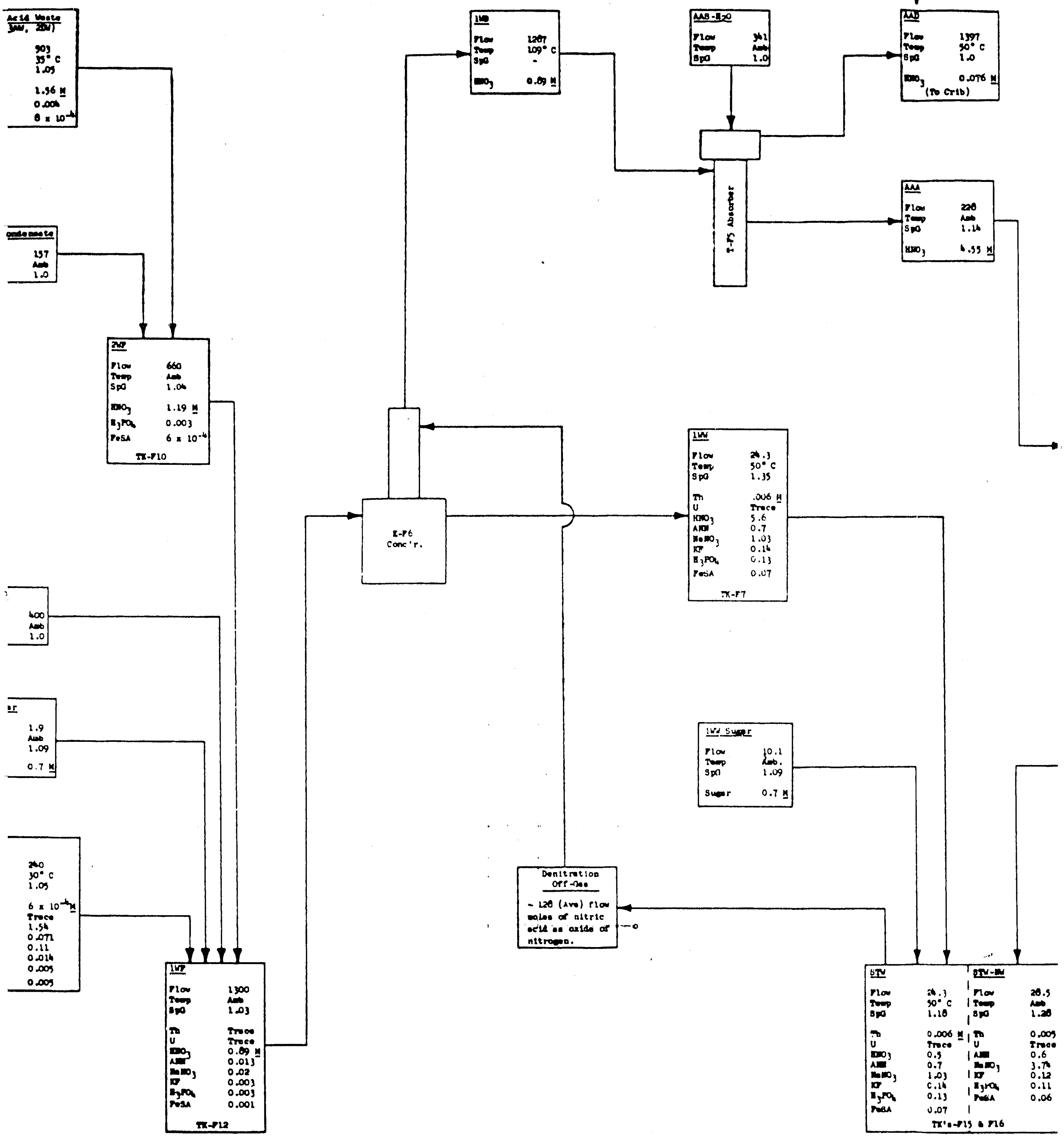
AT-809

WASTE CONCENTRATION AND ACID RECOVERY



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SECTION 3

PROCESS CONTROL

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3. PROCESS CONTROL

3.0 Introduction

Section 2 of this document presents the chemical flowsheet that is recommended for Purex Plant, Thorium - Uranium-233 separations. Section 4 presents the process specifications that must be followed in order to insure safe processing and the production of products of adequate quality. This section, Process Control, discusses the requirements for practical application of flowsheet and specifications to the Purex Plant processing system.

The process control recommendations can be grouped into the following general functions that are required in the plant to successful thorium processing:

(a) Requirements for the control of product cross-contamination are discussed and a system for control is proposed. A plan for flushing and recommendations for isolating processing systems, such as by blanking of process routings, are included.

(b) Discussions are presented of the important elements of process control that are required for specific application of the thorium chemical flowsheet to the Purex Plant processing complex. In addition, product rework is discussed, and a sample schedule is recommended.

(c) Recommendations with regard to the control of hazards are included, as appropriate, throughout this section. In addition, certain subsections (e.g., 3.10 - Chemical Hazards) are specific to hazards control.

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3.1 Cross-Contamination and Flushing

Contamination of the uranium-233 and thorium product streams could result from any of several sources. The precautions recommended for preventing the accidental charging of normal irradiated uranium slugs and the inadvertent transfer into the process of stored solution containing normal irradiated uranium and plutonium are described in Sections 3.2 and 3.9, respectively. The problem of contamination from residual uranium and plutonium in the equipment after completion of normal processing (and, to a lesser extent, thorium and uranium-233 contamination after completion of a thorium processing campaign) is minimized by special flushing of all equipment pieces. The following discussion is concerned with this latter aspect of contamination control and presents the recommended criteria and over-all plan for flushing the plant to the required degree.

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3.1.1 Bases and Objectives for Flushing

The flushing requirements are based on a combination of the product purity goals proposed in Section 4, the capabilities for blending product batches, and previous experience with respect to the effectiveness of various flushing techniques. The limits recommended in Section 4 for uranium isotopic and plutonium impurities in the uranium-233 and thorium product streams are as follows:

U Isotopes in U-233	-	3.9%
U Isotopes in Thorium	-	10 ppm
Pu in U-233	-	0.1%
Pu in Thorium	-	10 ppm

No facilities are available for blending large quantities of uranium-233 solution of different isotopic impurities, except by rework through the plant. The thorium product (TNT) solution, however, will be stored in large tanks in the WR Vault and thus blending into one or two large batches is feasible. The plant experience with respect to the effectiveness of various flush methods and solutions is discussed in detail later in this section.

The steady-state inventory of uranium-233 in the final U-233 concentration and loadout (N-Cell) equipment is estimated at 25 Kg (~14 Kg in the N6 Concentrator and ~11 Kg in the N7 Receiver and/or Loadout Tank). Most of the residual U-238 from those systems which will process U-233 will probably be "smeared" over the first 40 Kg of U-233 product. The 3.9 percent isotopic impurity limit on the U-233 product, combined with the above condition, limits the quantity of normal

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irradiated uranium remaining in the acid recovery, dissolvers and feed preparation, and U-233 processing systems to 3.4 pounds. It is further recommended that a safety factor of about 1.5 be applied to this limit to assure meeting the purity goals and to permit recycle of thorium product from TK-K6 to TK-E6 (in the event it is necessary to rework large amounts of U-233 product). Application of this safety factor results in an allowable average U-238 concentration in the 3AF stream of about 4×10^{-4} pounds per gallon. Application of the same bases to the plutonium contamination in the uranium-233 product downstream of the 2A Column (T-J22) gives a Pu limit of 40 grams, total. This corresponds to an allowable 3AF Pu concentration of 5×10^{8} c/min/gal. (Plutonium will be effectively separated from the product streams in the HA and 2A Columns by addition of ferrous sulfamate.)

Allowing for the blending capability for thorium product in the storage tanks, the thorium product purity goal limits the quantity of normal irradiated uranium in the thorium processing equipment downstream of the 1BS Column to three pounds (for 150 tons of thorium) or 4×10^{-5} lb/gal in the TNT product (TK-K6). The corresponding plutonium limit is 0.003 pound, or 1.36 grams, total. However, since the plutonium content in the K-Cell equipment is normally very low, compared with uranium-238, the residual plutonium will be reduced to well below the required limit by the flushing which is planned.

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3.1.2 General Flushing Plan and Goals

Comprehensive planning and coordination will be required to minimize scheduling delays and accomplish, within a reasonable period of time, the degree of flushing required to achieve the product purity requirements. This applies especially to the flushing prior to a thorium campaign, and also, to a lesser extent, to the post-campaign flushing. The general plan for flushing the Purex Plant both prior to and after the thorium campaign is outlined in this section. The following phase-by-phase summary describes the principal flushing operations and required timing and integration for the flushing program. When the flushing program is completed as outlined, the residual uranium-238 is expected to be distributed approximately as shown in the following table:

Dissolvers and Auxiliaries	$\sim$1.4 lbs
Acid Recovery System	$<$ 0.1 lb
Feed Preparation and E Cell	$<$ 0.3 lb
Solvent Extraction and U-233 Loadout (Excluding T-J7, E-J8, and K Cell)	$<$ 0.5 lb
Final Thorium Cycle (T-J7, E-J8, and K Cell)	$<$ 3 lbs

Phase 1. Purging of Acid Recovery System

Preparation of the Purex Acid Recovery System for a thorium campaign involves no flushing; rather, addition of UO_3 acid (containing uranium) is terminated and residual uranium is purged from the system. Two to three weeks prior to shutdown of normal processing, addition of UO_3-recovered acid to the Fractionator Feed Tank (TK-U5) should be stopped

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and the line blanked. The use of UO_3 acid for uranium-plutonium processing may be continued provided the following conditions are met:

(a) In all addition systems ("Christmas Trees"), where either Purex Recovered or UO_3 Recovered acid can be added, blank(s) should be installed in the system(s) not in use to insure against cross-contamination because of leaky valves.

(b) UO_3 acid may be used in the dissolvers for uranium metal dissolution provided the concentration is ≥ 48 wt. percent, to insure against dissolving to acid deficiency with standard procedures. To guard against receiving acid < 48 percent, the Uranium Oxide Operation should be notified to adopt special procedures to permit sampling each batch of acid before it is shipped.

To aid in purging uranium from the system, the U1 and U2 receiver tanks should be pumped to the minimum heel each time before switching receivers and the samplers should be well circulated each time. In addition, special effort should be made to insure that the Recovered Acid Header is well purged of uranium-containing acid and that no "dead pockets" are left at ends of lines, etc.

Return of the Purex Acid Recovery System to normal processing after the thorium run involves merely removing the necessary blanks to permit addition of UO_3 Recovered acid to TK-U5.

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Phase 2. Shutdown Preparations

Generally, normal shutdown preparations and procedures will be used, except that special effort should be made to purge uranium from the system. Special considerations include:

(a) Special heel cuts in the dissolvers; solution to be processed through plant prior to shutdown.

(b) Minimize uranium and plutonium inventory in Backcycle Waste System by keeping losses at the minimum levels.

(c) Optional - after the plutonium is fairly well purged from the system (< 3 AT $\leq 1 \times 10^{10}$ c/min/gal), the 2BP may be routed to the L-Cell Package to permit an early start in the stripping and flushing of N Cell.

Phase 3. Flushing of Dissolvers and Feed Preparation System

This phase should commence immediately following the final heel cuts. The initial flush solutions are concentrated and stored, with the 3WB, in TK-D3/D4. Subsequent flushes are processed through the Waste Treatment System. Dissolver flush solutions are also used to flush the D and E-Cell vessels. The latter portion of this flushing will be performed concurrently with the Solvent Extraction System flushing, so it will be necessary to integrate these flushes to expedite processing of the solutions through F Cell. The "Head-End" flushes will be routed to TK-F10 (or TK-F12) via the TK-F8 to TK-D5 route until all recoverable solutions have been transferred to storage.

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Phase 4. Flushing of L-Cell Package, N-Cell, and PR Room

This phase of flushing may, optionally, commence during the shut-down of normal processing. Flushing of N-Cell consists of removal of all plutonium from the TK-N1, E-N6, and TK-N7 vessels and all interconnecting piping. In addition, the hood sumps should be flushed as free of plutonium as practicable. These solutions will be routed to TK-E6 until all recoverable solution has been transferred to storage. They are then routed to TK-F18 for disposal. Flushing of the L-Cell Package, sump, and PR Room will remove the bulk of the plutonium. However, since it is not planned that these facilities will be used for product handling, it is not necessary to flush them extensively.

Phase 5. Flushing of Solvent Extraction System

Flushing of the Solvent Extraction System should commence immediately following shutdown from normal processing. This phase of the over-all flushing program consists of:

(a) Product recovery from all vessels.
(b) Dilute acid flushing of entire system, including transfer lines.
(c) Decontamination flushing of certain vessels.
(d) Final flushing and operability run.

Flushes should be made of all vessels, possibly excepting the H2, H3, J22, J23, K2, and L1 columns, for removal of recoverable quantities of product. These flush solutions are concentrated in E-H4 and transferred to TK-D3/D4 for storage. Flushing of the UNH Transfer System (both lines) should also be performed at this time.

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The dilute acid flushes are intended to remove the final significant traces of product from the system. All interconnecting lines, as well as samplers, should be flushed at this time to insure elimination of "pockets". Routing of these flush solutions to TK-F12 and E-F6 for acid recovery must be integrated with Head-End flushing operations. The solution analyses may be used as guides for determining the progress of this flush. However, since the analytical data are not very accurate at the expected low uranium and plutonium levels, they should not be relied on too strongly. Two separate, complete flushes of each equipment piece should be adequate.

To conserve both time and waste storage space, only those equipment pieces which contain high levels of contamination or quantities of crud or scale should be given decontamination flushes. Because decontamination flushes are notorious time consumers, special attention should be given to integrating these flushes, both in AMU and Waste Treatment operations. A single sequence (caustic-tartaric, nitric-oxalic) should suffice. In addition, it may be desirable to flush the H2 and H3 Columns with decontaminating solution immediately after shutdown to prevent spread of fission product contamination to downstream vessels during the acid flushes.

The final flushing of the Solvent Extraction System will be combined with a "cold" operability run using dilute (5 - 10%) nitric acid solutions and 30 percent TBP solvent.

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[redacted]

Phase 6. Final Flushing and Operability Run

The final flush of the plant is combined with a "cold" operability run in which all systems should be checked. The "cold" run should be made at approximately flowsheet conditions and the flow rates planned for the "hot" run, except that the simulated HAF will be dilute nitric acid. The "cold" run should continue until the uranium and plutonium analyses of critical streams (discussed later) are within the recommended limits and all systems are operating satisfactorily.

Phase 7. Terminal Flush of Plant

The terminal flush of the plant, at the completion of the thorium campaign, will be very similar to the Phase 6 flushing, except the dissolvers will require extensive heel cuts. Flushing of the remainder of the system will be accomplished with dilute nitric acid. This flush is estimated to require about three days.

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3.1.3 General Flushing Criteria

The following precautions and criteria apply generally to the entire flushing program.

(a) All routings to and from each vessel should be either flushed or removed if there is a possibility that they might be contaminated with normal uranium.

(b) Unless otherwise specified, all chemical flush solutions should be digested in each vessel for at least two hours, with agitation, at a temperature of 40 - 60° C.

(c) The samplers in all vessels and streams that will be used for thorium processing must be operable and free of uranium-238. To expedite flushing, all samplers not routinely used (or inoperable samplers) should be thoroughly checked and repaired prior to shutdown. All samplers should be thoroughly flushed by circulating all acid and water flush solutions through them whenever the solution temperatures are below 60° C.

(d) Samples should be obtained (for U, HNO_3, and AT analyses) from every vessel after completion of the flushing (digestion) period. In addition, fluoride and iron analyses should be requested for samples of nitric-fluoride solutions.

The transfer of flush solutions should not be held up because of an inoperable sampler. However, the cause of the trouble should be determined and remedied without delay.

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(e) No flush solution containing fluoride should be heated above 30° C, unless the Al/F mole ratio is at least 4.0 or the fluoride concentration is $\leq$ 0.001 *M*.

(f) To minimize the volume sent to underground storage, high-acid or high-salt flush solutions should be reused as much as possible.

(g) Flush solutions containing aluminum should be limited to a maximum concentration of 1.0 *M* Al (3.13 lb/gal ANN). If the concentrated waste is greater than 0.5 *M* ANN in aluminum, a "reverse strike" (addition of the waste to the caustic) is required for neutralization.

(h) Special precautions should be taken to assure that all non-boiling waste solutions containing greater than 0.25 *M* aluminum are neutralized to a pH of $\geq$ 12 before transferring them to underground storage.

(i) Concentrated flushing wastes should be routed to non-boiling waste storage if the total fission product activity (ZrNb-95, Ru-103, RuRh-106, and Ce-144) is less than 3.5×10^7 uc/gal.

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3.1.4 Flushing Sequence and Compositions

A. Acid Recovery System

Uranium is purged from the Purex Acid Recovery System by stopping the addition of UO_3 Recovered Acid to the Purex Acid Recovery System (normally TK-U5). Since the displacement time is about one day in this system and the minimum number of displacements for satisfactory removal of uranium from TK-U5, T-U6, TK-U1, and TK-U2 system is on the order of four to six displacements, it is suggested that the addition of UO_3 acid to the Purex Acid Recovery System (TK-U5) be terminated at least 10 days prior to the end of the normal uranium campaign which precedes the thorium campaign.

The use of UO_3 acid may be continued in TK-E6, the 1BXP-HNO_3, and the 2DF-HNO_3 streams during normal uranium processing. UO_3 acid may also be used in the Purex dissolvers during normal uranium processing provided adequate precautions are taken to prevent reaching an acid deficient condition during the dissolution of normal uranium. The recommended procedure involves sampling of a uniform batch of UO_3 acid for HNO_3 analysis (and butting, if necessary) to insure that the HNO_3 content is 48 wt. percent before it is shipped to the Purex Plant.

B. Dissolver Heel Removal

The flow pattern recommended for removing and recovering normal uranium from the dissolver system is shown in Figure 3.1-I.

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Initial removal of the heel from the dissolvers should be performed according to the standard procedure, except that the initial charge of acid to the annular dissolvers should be increased to 600 gallons, in order to submerge the slug grid in the annular dissolver. The initial heel removal solution should be processed through solvent extraction prior to plant shutdown.

The final heel removal should also use about 600 gallons of acid (Purex Recovered) per cut. Since the final heel-cut solution will be stored (rather than processed prior to plant shutdown), the final heel cutting operation should continue, using the same acid, until no further specific-gravity rise is detected after 16 hours of digestion, or until analytical results show less than one pound uranium dissolved per hour of digestion. The heel removal operation should be completed by the time the Solvent Extraction System is shut down completely.

C. Final Head-End Flushing

1. General

The over-all flow pattern and disposition of the flush solutions is presented in Figure 3.1-II. The caustic flush solutions are routed to non-boiling waste storage via the E-Cell vessels (TK-E3 → G-E2/E4 → TK-E5 → non-boiling waste storage). The acid flushes are routed to E-F6 (for acid recovery) via the normal HAF feed route and/or the E Cell centrifuge system. The time required to flush the

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Head-End System is estimated at eight to ten days.

2. Dissolvers and Associated Equipment

Flushing of the dissolver vessels should commence as soon as the heel cutting is completed. The suggested flushing sequence, outlined below, is based on experience gained during the flushing program for the special Purex test run during the winter of 1964, 1965 and at Savannah River. In each instance, dissolution of residual uranium was greatly enhanced by a caustic digestion followed by a nitric-fluoride digestion. It is not known for certain whether the fluoride ion or the digestion with caustic was responsible for the increased dissolution rate. This question should be resolved, however, by the results of flushes four and five. Since it may be necessary to repeat part of the flushing sequence or introduce special flushes for certain equipment pieces, depending on the initial results, the last flush listed should not be construed as the final one.

Flush No.	Solution
1	Dilute acid ($\sim$10% HNO_3) boilup
2	Caustic-sodium nitrate*
3	Water rinse
4	Nitric flush
5	Nitric-fluoride flush
6	Water rinse
7	Caustic-tartaric flush
8	Water rinse
9	Nitric-fluoride flush; condensate routed to TK-F10 or crib
10	Nitric-fluoride flush; condensate refluxed to dissolver

*This flush is to remove the last trace of aluminum coatings.

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The following are the suggested compositions, volumes, digestion times, and dispositions of the flush solutions:

Dilute Acid: The initial dilute acid flush consists of filling the dissolver to within 10 per cent of overflow with 10 per cent nitric acid and alternately boiling (under total reflux) and cooling for several cycles over a 24-hour period. This deep flush should also flush the vapor line and downdraft condenser. If this flush dissolves more than ten pounds of uranium during the first 24 hours, it should be continued, using the same solution, until the uranium dissolution rate is less than 0.4 pounds per hour. If this flush solution contains sufficient product to be economically recoverable, it should be routed to E-H4 Concentrator. Otherwise, it should be routed to E-F6 Concentrator for acid recovery.

Caustic-Sodium Nitrate Flush: This will be a standard coating removal operation, except that only 400 gallons of sodium nitrate are required (just enough to cover any end pieces on the support grid) and the caustic can be added at a faster rate (up to 100 lb/min). After addition of 3000 pounds of 50 per cent caustic and a two-hour digestion, a sufficient quantity of 10 per cent caustic should be added to cover the coils and the solution boiled for an additional three hours. After completion of the five-hour digestion step, the caustic

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solutions should be routed to non-boiling waste storage via E-Cell. (Note: To avoid contaminating TK-E1, this flush solution and the centrifuge cleanout solutions should be routed from the centrifuges to TK-E5).

Water Rinses: Water rinses of about 1000 gallons each should be made after each chemical flush.

Nitric-Acid and Nitric-Fluoride Flushes: These flushes will require about 4800 gallons of 57 per cent nitric acid for each annular dissolver. The nitric fluoride solutions will require, in addition, 0.1 M ANN and 0.025 M fluoride. The flush solutions should be digested for about 16 hours, cooled and sampled, then digested another 16 hours, cooled and sampled. The digestion should be at the maximum air sparge rate, the maximum boilup rate the off-gas system will permit, and, generally, under total reflux of the condensate. The C3 condensate should occasionally be routed to TK-F10 or TK-F3 to flush the lines.

Caustic Tartaric Flushes: These flushes consist of about 2000 gallons of ten per cent caustic (NaOH) and two per cent tartaric acid. They are intended primarily as "pretreatment" flushes and to dissolve any scale that might be present. (Flushing results for the special test run indicated much higher uranium dissolution rates for the nitric-fluoride flushes immediately following a digestion with caustic solution.) The caustic-tartaric flushes should be digested for four to eight hours, then transferred to TK-E3. The

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caustic-tartaric solution should be butted to 4800 gallons with fresh ten per cent caustic for flushing the E-Cell vessels.

3. Metal Storage and Feed Adjustment Tanks

Flushing of the metal storage and feed make-up tanks should continue until the TK-D5 and TK-H1 uranium concentrations are $< 1 \times 10^{-5}$ and $< 8 \times 10^{-5}$ lb/gallon, respectively. Flushing of these tanks (Tanks D1, D5, E6 and H1) is intended primarily to remove residual uranium. The uranium heels in these tanks will be partially removed during shutdown by transfer of the final dissolver heel cuts through the tanks. Additional heel dilution will be accomplished when the final concentrated recoverable flush solutions are transferred back to TK-D3/D4 from TK-J1. These flush solutions should be routed from TK-J1 to TK-H1 to TK-E6 to TK-D3/D4 for interim storage.

After the concentrated acidic product removal solutions (Phase D-1 of flushing) have been transferred through Tanks H1 and E6 to TK-D3/D4, they should be sufficiently free of uranium that all subsequent flush solutions can be routed to E-F6 for acid recovery, as shown in Figure 3.1-IIa. Routing of the dissolver rinse and nitric-fluoride flush solutions through the feed storage and make-up tanks should clean them to the desired levels. However, to assure the most effective flushing of these tanks, they should be filled to the maximum with the nitric-fluoride flush solutions and the flush solutions

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should be digested for at least two hours in each tank at 40 to 50° C with tank samplers circulating during the entire digestion period. Samples should be obtained from each tank at the end of each digestion period. In addition, each tank should be completely emptied between each batch of flush solution.

4. Fission Product Recovery

Flushing of the E-Cell (TK's E1, E3, and E5 and centrifuges G-E2 and G-E4) is primarily for the removal of fission products. The uranium contamination in these vessels should be less than the required limit prior to flushing. The proposed flow pattern for flushing the E-Cell vessels is shown in Figure 3.1-IIb.

Following termination of the fission product recovery activities and before routing any dissolver flush solutions through them, the E-Cell vessels should be flushed with water and dilute nitric acid. The suggested procedure for these flushes follows:

1. Fill TK-E1 to maximum with 5% HNO_3, heat to 50 to 60° C, agitate, and circulate the sampler for one hour.
2. Jet the 5% HNO_3 from TK-E1 to TK-F18. Flush the centrifuges G-E2 and E4, with 5% HNO_3 to TK-E5. (Note: Transfer of flush solutions from the centrifuges to TK-E1 should be avoided until the centrifuges are relatively clean to avoid contaminating TK-E1.)

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3. Fill TK-E3 to maximum with 5% HNO_3, heat to 50 to 60° C, agitate, and recirculate the sampler for one hour.
4. Jet TK-E3 contents to TK-E5 via the centrifuges G-E2 and G-E4.

5. Digest the flush solution in TK-E5 for an hour with the sampler circulating.
6. Jet TK-E5 to TK-F18 and sample for disposal.

D. Solvent Extraction System

The flushing program for the Solvent Extraction System is comprised of the following phases:

1. Product Recovery (removal of bulk quantities of product from all vessels).

2. Final Product Removal (dilute acid flushing of entire system, including transfer lines, to remove final traces of uranium-238).

3. Decontamination Flushing (of certain vessels).

4. Final Flushing (combined with the "cold" operability run for all systems).

The suggested sequence and routings for flushing the Solvent Extraction System are shown in Figures 3.1-III, 3.1-IVa and 3.1-IVb.

1. Product Recovery

Acid flushes should be made of all vessels, including sumps, which might contain recoverable quantities of uranium or

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plutonium. Samples taken near the completion of the stripping and solvent displacement operations at shutdown would be helpful in determining which vessels could be excepted from this flush. Possible exceptions include the H2, H3, J22, J23, K2 and L1 columns.

The suggested flush solutions are five to ten per cent nitric acid for the partition columns, TK-J21, and the L and N-Cell equipment, and dilute (0.01 M) nitric acid for all other vessels and lines. The product recovery flushes should continue until the product values in any particular unconcentrated "batch" of solution are low enough to be discarded (e. g., the J7 → J8 → K1 flush should continue until the K1 uranium analysis is low enough to permit discard of a full K1 batch). These flushes should be transferred batchwise from vessel to vessel as much as practicable. All product recovery flush solutions, except those originating in L Cell, N Cell, or the PR Room are concentrated in E-H4 to about 10 M HNO_3 for storage in TK-D3/D4. All transfer routes which are to be eliminated (i.e., 3WB to T-H2, HSR to T-H2, 1BU to T-J7) and all N Cell and PR Room equipment scheduled for replacement, in addition to all transfer lines in N Cell and the PR Room, should be well flushed during this phase of flushing, as many of them will be removed before Phase 2 flushing. All columns should be pulsed, pumps, agitators, and jets should be operated, and samplers should be recirculated during these flushes. The

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solution temperatures should be maintained at 50 to 65° C. The UNH transfer and loadout facilities should be flushed at this time. The entire route, from TK-K6 to the trailer and from the trailer to TK-K1, should be well flushed with water. The initial flushes should be routed to the P tanks and the final flushes should terminate in TK-K6 (or TK-K1).

2. Dilute Acid Flush

The dilute acid flush of the solvent extraction system is intended to remove the final significant traces of uranium and plutonium from the system. While the product removed by this flushing can be recovered, the quantity involved should be small enough to permit throwaway. This flush should not be made until the major routing changes have been completed. This phase of the flushing, shown in Figure 3.1-IV, will also use dilute nitric, but the flush solutions will be added via a greater number of different lines. All flush solutions, except those from L-Cell will be accumulated in E-F6, concentrated, and sent to UGS. L-Cell and PR Room flushes are routed to TK-E6, then to TK-D3/D4. (Note: TK-E6 must be at least half full of solution to receive rework plutonium solution from TK-L11. Therefore, some of the Product Recovery flushes should be left in TK-E6 until the L-Cell, N-Cell, and PR Room flushes can be discarded). Two separate flushes, in which the columns or tanks are filled to overflow and pulsed or agitated for one hour should suffice. In cases where the flow is continuous, at least four throughputs of flush solution should be made.

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3. Decontamination Flushes

Only those equipment pieces which contain high levels of contamination or quantities of crud or scale will be flushed in this phase. These include the H2, H3, and J22 columns, the E-H4 concentrator, and the J1 and J21 tanks and interconnecting piping. A single sequence of standard decontamiation flushes (caustic-tartaric followed by nitric-oxalic) is suggested. In addition, it might be necessary to give E-H4 and TK-J1 a high-velocity water flush to remove undissolved scale and crud*. This can be accomplished by connecting a flex jumper to one of the nozzles supplying raw water to the E-H4-2 condenser and overflowing the vessels to the sump.

A preliminary decontamination flush of the T-H2 and T-H3 Columns might be desirable to prevent fission products and crud in these columns from contaminating subsequent vessels. This flush would be made immediately after shutdown and would follow the normal routing for flushes of the HA and HS Columns, via TK-324 → T-H3 → T-H2 → TK-F7 → UGS.

Since the HSR jumper should have been removed by the time the major portion of the decontamination flushing commences, the suggested routing of the flush solutions is as follows:

(a) T-H3 → E-H4 → TK-J1 → TK-F10 → TK-F12 → E-F6 → UGS;

(b) T-H2 → TK-F10 → etc.;

(c) TK-J21 → TK-F10 → etc.;

(d) T-J22 → TK-F10 → etc.

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* At the time of this publication, the chemical flushing of E-H4 using a special procedure involving acid-fluoride is under consideration.

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These solutions should be moved batchwise between vessels as much as practicable. This phase should not require more than two days, as some (or all) of the flushes can proceed concurrently.

4. Final Flush

The final flushing of the Solvent Extraction System will be combined with a "Cold" operability run using dilute (5 - 10%) nitric acid solutions and 30% TBP solvent. During the cold run, all streams should be flowing at the planned rates and temperatues and all pulsers operating at stable frequencies. Care should be taken in adjusting frequencies, as some columns may flood at lower frequencies and some may flood at higher frequencies than normal.

E. Final Flushing and Operability Run

The final flush will be of the entire process system, including dissolvers, feed preparation, solvent extraction, product concentration, and waste treatment (except U Cell). This flush will consist essentially of a "cold" run using five to ten per cent nitric acid for HAF and 30 per cent TBP solvent. All other addition streams should be of flowsheet composition. The run should be made at the same conditions of flow rate, temperature, etc. as scheduled for the thorium campaign*. All systems, including instruments, "cold" addition lines, mechanical equipment, jets, and samplers, should be checked for operability at this time. Initially, the dilute acid for the HAF should

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*Except that volume reduction in the E-J8 and E-K4 concentrators should be limited to about 50 per cent.

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originate in the dissolvers and pass through all process equipment, including samplers. After the flush solution in TK-D5 is $< 1 \times 10^{-5}$ lb/gal uranium, the simulated HAF can be made up in TK-E6 and charging of the dissolvers can commence. The simulated product stream (3UC) should be drained to the N-Cell sump, transferred to TK-L11, and either recycled to TK-E6 or, preferably, drained to S-LD and transferred to S-LA to TK-F18 to underground storage. The acid in TK-K6 can be jetted to TK-F13, TK-F10, or TK-F7. The simulated 3AF in TK-J5 should be routed to TK-F10 via T-L1 until the uranium concentration is less than 4×10^{-4} lb/gal (corresponding to three per cent isotopic impurity in the U-233).

The time required for this phase of flushing (and testing) is estimated at three days.

F. Terminal Flushing

The terminal flushing program, at the conclusion of the thorium campaign, will be very similar to the final pre-campaign flushing (Phase 6), except that all aqueous streams can be composed of dilute nitric acid. Dissolver flushing will consist of extensive heel cuts using the same solution composition as during regular thoria dissolution (11 $\underline{M}$ HNO_3, 0.025 $\underline{M}$ F^-, and 0.10 $\underline{M}$ Al). These should continue until the thoria dissolution rate is less than 50 grams per hour. The time required for this flushing is estimated at three days.

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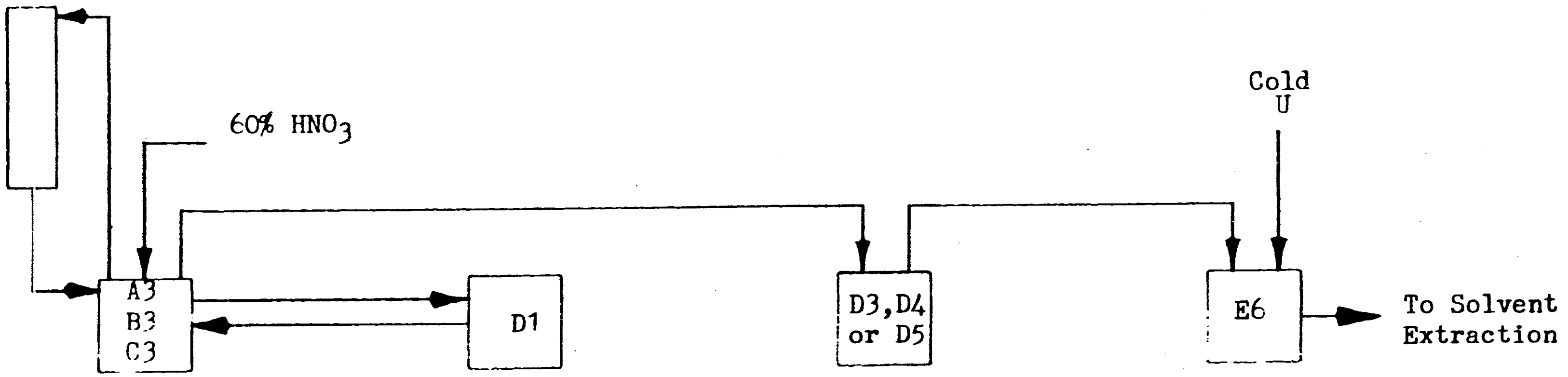
a. Initial Heel Cuts



b. Final Heel Removal

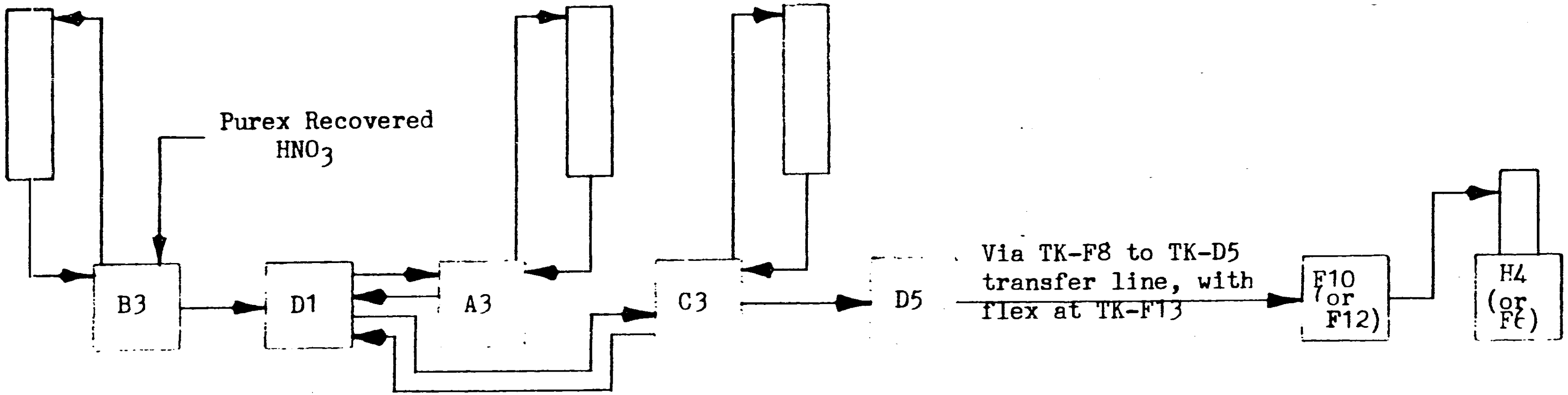


FIGURE 3.1-I
HEAD-END FLUSHING
BULK REMOVAL OF URANIUM HEELS

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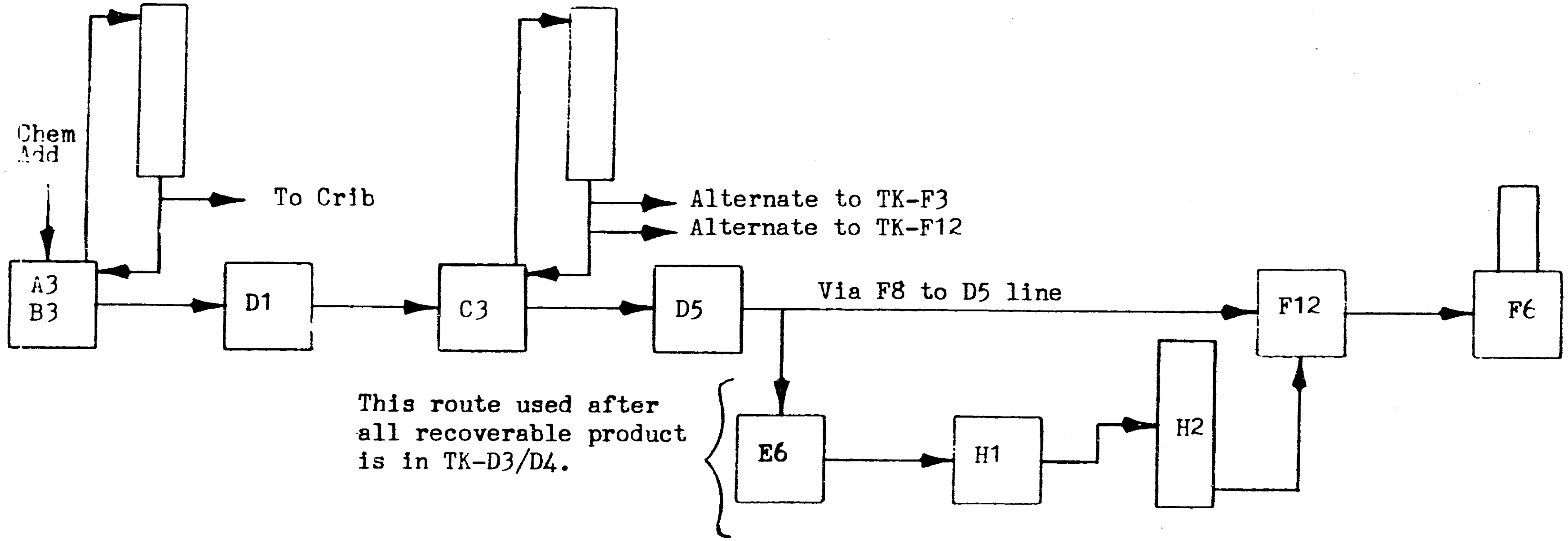
a. Feed Storage and Makeup Vessels



b. E-Cell Vessels

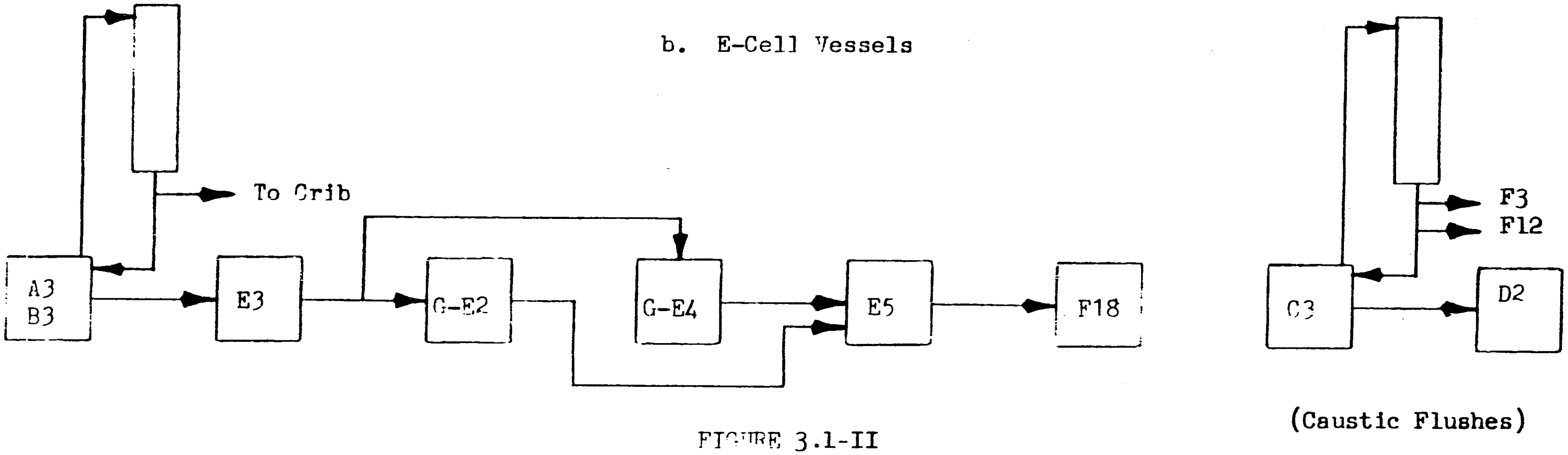


FIGURE 3.1-II
FINAL HEAD-END FLUSHING

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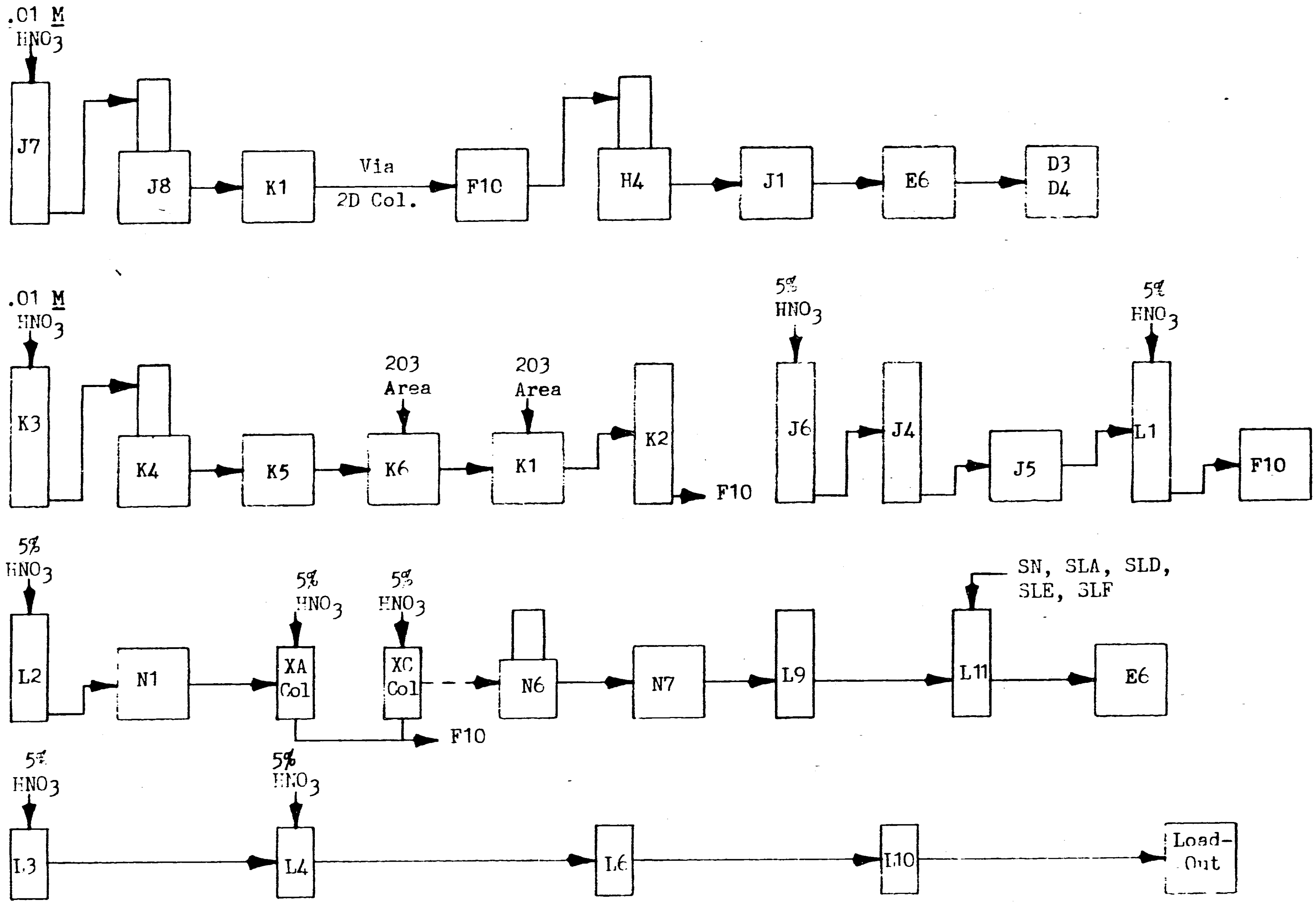

FIGURE 3.1-III
SOLVENT EXTRACTION SYSTEM FLUSHING
Phase 1. Bulk Removal of Product

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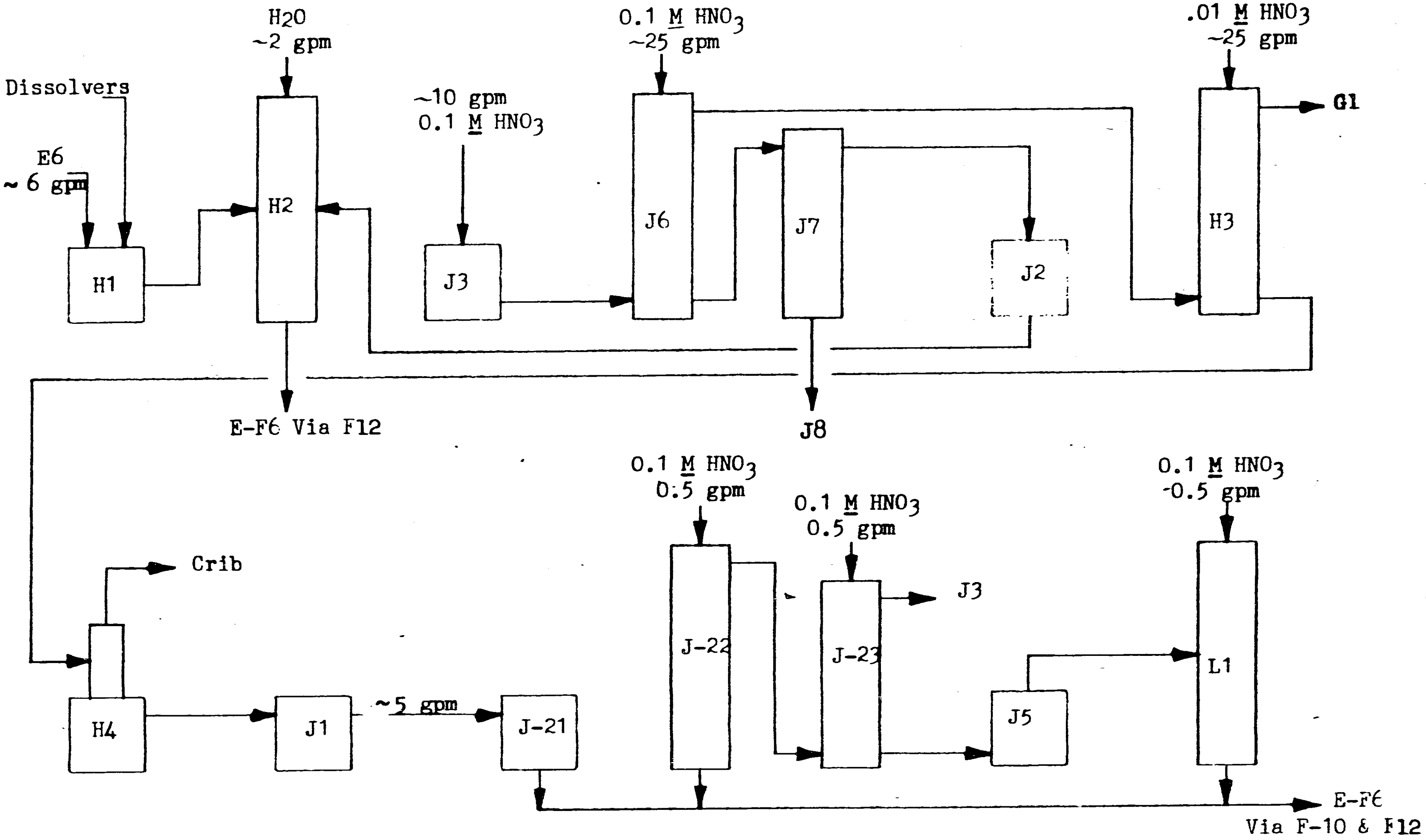

FIGURE 3.1-IV-a
SOLVENT EXTRACTION SYSTEM FLUSHING
Phase 2. Dilute Acid Flushes

Note: Omit fluoride from E6 Solutions until recoverable quantities of U & Pu have been accumulated in E-F6.

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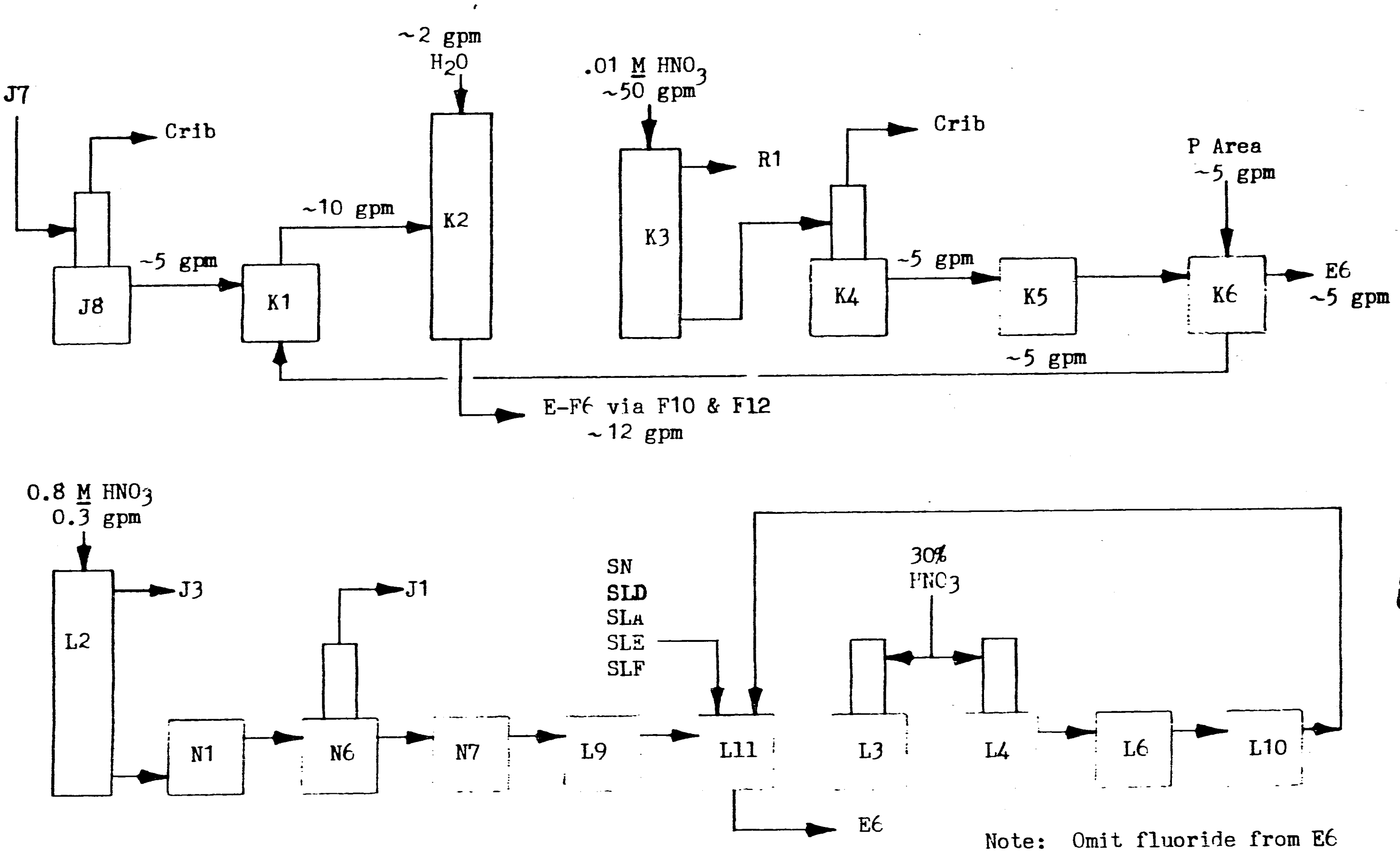

FIGURE 3.1-IV-b
SOLVENT EXTRACTION SYSTEM FLUSHING
Phase 2. Dilute Acid Flushes

Note: Omit fluoride from E6 solutions until recoverable quantities of Uranium and Plutonium have been accumulated in E-F6.

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3.2 Feed Preparation

3.2.1 Charging and Can Removal

The amount of thoria which can be charged to a dissolver is restricted to the maximum contained in 9 buckets of irradiated thoria target elements. This is about the maximum quantity which can be dissolved in one thoria dissolution cut in a reasonable time. A larger charge, requiring more than one cut, cannot be made because the pile of thoria in the dissolver inner annulus after removing the aluminum cans would block the drainage of the can removal waste solution to the outer annulus.

The amount of thoria and aluminum expected for different charge sizes is shown in Table 3.2-I. With a maximum charge of 9 buckets, the maximum amount of thoria expected per charge is 4.1 tons (8200 pounds), and the maximum quantity of aluminum is 580 pounds. At the beginning of the thorium processing run, it is recommended that the charge size be limited to 7 buckets. If no operating problems are encountered, later increases to 8 buckets and to 9 buckets are suggested.

During thorium processing, precautions must be taken to prevent charging irradiated natural uranium to a dissolver. Charging a bucket of irradiated natural uranium would grossly contaminate the uranium-233 product. In addition, the plutonium-239 introduced would present a nuclear safety problem, since it would polymerize and precipitate during the acid boil-off operation. Figure 3.2-I shows the stacking configuration that is used to distinguish thoria elements. As an additional precaution against charging irradiated natural uranium, the tunnel gamma monitor should be adjusted to alarm at a setting that will indicate the presence of a uranium bucket.

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The critical mass specification requires that the combined mass of uranium-233 and protactinium-233 in the irradiated thoria must be less than 2200 grams per ton of thorium. Although no irradiations are scheduled to have any yields nearly this high, the IPD shipping records should be checked before each charge.

Iodine-131 can be released from the Purex plant at a rate not to exceed 1.8 curies per week. Purex experience indicates that 100 curies I-131 per day can be charged without exceeding this limit. This allows an average iodine-131 content in the thoria of about 10 curies per ton of thorium. The iodine-131 content of irradiated thoria as a function of cooling time and total 233 content, is shown in Figures 3.2-II and 3.2-III. For the 233 yields scheduled (1300 gm/ton ThO_2 maximum from core loadings and 1200 gm/ton ThO_2 from fringe loadings), a cooling time of 90 days or greater is adequate.

Before thoria elements are dumped into a dissolver, sufficient volume of sodium nitrate solution or water should be present in order that the dumped elements will be submerged. A 50% void volume in the pile of dumped elements should be assumed. If the crane operator can see elements above the solution level in the dissolver after charging, additional water should be added. Although thoria is not pyrophoric as is uranium metal, it is still recommended, as a safety precaution, that the elements be covered whenever practical.

The can removal should be made in the same manner used for uranium processing. The hydrogen content of the off-gases should be kept below 2.0 volume percent and the off-gas temperature from the

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down draft condenser should be maintained above 45° C in order to prevent a hydrogen or ammonia explosion. The ammonia scrubbers should be operated with at least 10 gpm flow of scrub water.

While the aluminum cans are being removed, the dissolver air-lift circulators should be operated as shown in Table 3.2-II. Once the thoria is released from the cans, the circulators must be operated continuously to avoid plugging. Steam sparging at a low rate will provide very little circulation but will prevent the lifters from plugging. In addition, during can removal both the inner and outer annulus circulators must be operated in a manner that will disperse little of the thoria to the outer annulus and in turn plug jet-out dip tubes. A high circulation rate of the dissolver solution by the outer annulus circulator is also to be avoided to prevent compacting the thoria bed in the inner annulus. When changing the lifters from steam to air operation or from air to steam operation, the switch should be made with as little interruption in sparger flow as possible.

In order to prevent erosion of the dissolver bottoms beneath the lifter-circulators, the impinging air velocity must be kept below 50 fps. This means that the air supplied to the inner annulus lifter-circulator must not exceed 300 scfm. The air supplied to the outer annulus lifter-circulator must not exceed 260 scfm.

The solution in the dissolver during can removal should not be concentrated. Ammonia scrub water should be added to the dissolver as necessary to maintain a constant volume. After the can removal

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waste has been cooled, the solution should be allowed to set the 15 minutes with minimum steam flow to both circulators in order to settle the thoria. With both circulators on minimum steam the dissolver contents can still be held below 100° F by using maximum cooling water flow to the dissolver coil.

The most important control features to be followed during charging and can removal are summarized in Table 3.2-III.

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3.2.2 Can Removal Waste Processing

In order to avoid precipitation of alumina in the can removal waste the sodium hydroxide content should be kept above 5 percent. However, even with the 5 percent sodium hydroxide concentration the stability of the can removal waste solution cannot be maintained indefinitely. For this reason a maximum retention time in the plant of 24 hours is recommended for this waste solution. If anything should happen to the centrifuge equipment, the waste solution should be sent to waste storage without centrifuging.

While centrifuging thoria from can removal waste solution, a centrifuge speed of 1200 rpm and a feed rate of 10 gpm is recommended.

Any thoria in the centrifuge bowl should be washed and slurried out with as little delay as possible. To prevent caking, the thoria should not be allowed to stand dry in the centrifuge bowl for a period of more than 10 minutes. After the thoria in the bowl is washed with caustic, it is rinsed with water before adding acid to avoid an exothermic heat of neutralization reaction. For the same reason, after the thoria is slurried from the centrifuge, the bowl is again rinsed with water before adding a caustic solution.

The thoria slurried to Tank-E1 should be agitated and digested in Tank-E1 at a temperature of about 70° C for 8 hours before jetting to Tank-D1. If another batch of can removal waste is ready to be centrifuged before a digestion period is finished, the centrifuging operation should proceed and Tank-E1 should be double batched.

The most important details of the process and nuclear control for coating waste processing are shown in Table 3.2-IV.

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3.2.3 Thoria Dissolution

After the can removal waste is jetted from the dissolver, 60% nitric acid is added to the dissolver. At least 10 moles of nitric acid per mole of thoria is recommended. The Tank-U1 and U2 analyses will indicate how much additional fluoride and aluminum are required to make the final dissolver solution 0.025 M fluoride and 0.1 M aluminum nitrate. A correction, however, must be applied to the aluminum nitrate required since a substantial quantity of aluminum will be contributed by the can removal waste solution heel in the dissolver. The dissolver inner circulator is changed from steam to air at a rate of 125 to 250 cfm. The outer circulator steam flow is set at a rate of about 500 pph, and the solution is boiled until the thoria heel is about 800 pounds.

After about 6 hours of boiling and at 3-hour intervals thereafter, the inner annulus circulator should be set on minimum air flow (125 cfm) and the outer annulus circulator switched to air at a rate of 150 cfm for 5 minutes. At the end of 5 minutes on air, the outer annulus circulator is switched back to steam at a rate of 500 pph, and the inner sparger air flow is returned to its previous flow. The time element of 5 minutes with respect to the outer circulator is important in order to prevent compaction of the thoria bed in the inner annulus. The purpose of the operation is to move thoria displaced to the outer annulus back to the inner annulus.

The air-lift circulator operation during thoria dissolution will make the dissolver specific gravity readings meaningless. Experimental data indicate however that the thoria should be essentially dissolved in approximately 12 hours. After about 10 hours of boiling and every 2 hours thereafter the inner annulus circulator should be put on steam at a

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rate of about 300 pph in order to obtain an accurate specific gravity reading. The amount of thoria dissolved as a function of the specific gravity can be estimed from Figure 3.2-IV.

Initially the dissolver solution will boil at 120° C; the boiling point temperature will fall to about 116° C as the dissolution proceeds. Figure 3.2-V shows the specific gravity of thorium-nitric acid solutions at 100° C. The specific gravity of these solutions decreases approximately 0.0012 specific gravity units per C°.

Table 3.2-V summarizes the dissolver lifter-circulator operation recommended during thoria dissolution. Other recommended control limitations to be followed during thoria dissolution are tabulated in Table 3.2-VI.

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3.2.4 Acid Boil-Off

In order to remove nitric acid from the thorium dissolver solution at a practical rate, it is necessary to first concentrate the solution to approximately 4.0 M thorium. After the solution has been concentrated, it is steam sparged and boiled at a constant volume until the remaining nitric acid is removed. During the constant volume boiling operation the boiling point of the dissolver solution will decrease from approximately 135° C to 123° C. With sufficient operating and analytical data, it may be possible to use the boiling point temperature in conjunction with specific gravity as an end point control guide. Before cooling and transferring the solution from C3 to Tank-D5, the thorium concentration is decreased to 1.5 M by the addition of demineralized water. The solution must be diluted before it is cooled to avoid precipitation of the thorium. The boiling point temperature of thorium-nitric acid solutions is shown in Figure 3.2-VI.

The control limitations recommended during the acid boil-off operation are summarized in Table 3.2-VII.

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3.2.5 Feed Make-Up

Demineralized water, 50 percent aqueous sodium hydroxide, and/or 57 percent aqueous nitric acid are added to Tank E6 to make the final solution.

Th	1.2 to 1.7 M
HNO_3	-0.05 M to -0.15 M

Rework thorium or uranium-233 may be added to Tank E6 from Tank-K6 or Tank-L11. All E6 solutions must contain less than 2200 grams protactinium plus uranium-233 per ton of thorium and the plutonium content of Tank E6 must not exceed 250 grams, in order to meet the critical mass specifications.

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TABLE 3.2-I

THORIA AND ALUMINUM EXPECTED PER DISSOLVER CHARGE

	Small Reactor Central Core	Small Reactor Fringe	K-Reactor Central Core	K-Reactor Fringe
Element length (in.)	8.865	8.865	8.865	8.865
Element diameter (in.)	1.445	1.445	1.600	1.480
Aluminum per element (lbs.)	0.23	0.23	0.26	0.24
Thoria per element (lbs.)	3.30	3.30	4.11	3.48
Elements per bucket	274	274	219	230
Aluminum per charge (lbs.)				
7-bucket	447	447	405	386
8-bucket	511	511	463	442
9-bucket	574	574	521	497
Thoria per charge (lbs.)				
7-bucket	6330	6330	6300	5600
8-bucket	7230	7230	7200	6400
9-bucket	8140	8140	8100	7200

[redacted]

[redacted]

TABLE-3.2-II

DISSOLVER LIFTER-CIRCULATOR OPERATION DURING CAN REMOVAL

Can Removal Operation	Inner Lifters	Outer Lifters
Heating sodium nitrate solution	Air (125 cfm to 250 cfm)	Steam (500 pph)
Sodium hydroxide addition	Steam (300 pph)	Steam (500 pph)
Digestion	Steam (300 pph)	Steam (500 pph)
Cooling solution	Air (125 cfm to 250 cfm)	Steam (500 pph)
Setting thoria	Steam (300 pph)	Steam (500 pph)
Jetting waste solution	Steam (300 pph)	Steam (500 pph)

[redacted]

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TABLE 3.2-III

SUMMARY OF PROCESSING AND NUCLEAR CONTROL FOR CHARGING AND CAN REMOVAL

Item	Control Limit	Control Method	Comments
Charge size	9 buckets (8200 lbs ThO_2, 580 lbs Al)	Direct count by crane operator	
Irradiated natural uranium bucket	Avoidance	Bucket stacking, tunnel gamma monitor	
U-233 & Pa-233 g/t	2200 gms U and Pa per ton Th	IPD records	
Pu in A3 or B3	250 gms Pu	IPD precautions, bucket stacking, tunnel gamma monitor	
I-131	1.8 curies/week to environs ($\geq$90 days cooling time)	IPD records	
Volume of $NaNO_3$ solution	Sufficient to cover all the thoria target elements charged	Observation by crane operator	
$NaNO_3$/Al mole ratio	1.00 mole $NaNO_3$ per mole Al	Charge size, nitrate solution concentration and volume	Approximately 142 lbs Al/ton ThO_2 maximum
NaOH/Al mole ratio	1.25 moles NaOH per mole Al	Charge size, sodium hydroxide solution concentration and volume	Approximately 142 lbs Al/ton ThO_2 maximum
NaOH addition rate	$\leq$30 lbs/min 50% solution	Flowmeter	
Digestion time	2 hours	Clock	
H_2 content of dissolver off-gases	2.0 volume %	Nitrate addition, dilution air, H_2 monitor	

[redacted]

[redacted]

TABLE 3.2-III (Cont'd)

Item	Control Limit	Control Method	Comments
NH_3 content of dissolver off-gases	8.0 volume %	Water flow to scrubber, NH_3 monitor	
Downdraft condenser off-gas temperature	≥45° C	Thermohm, water flow to condenser	
Ammonia scrubber scrub water flow rate	≥10 gpm	Flowmeter	
Silver reactor influent gas temp.	350 to 400° F	Thermohm, electric heater	
Off-gas routing	Direct to 219-A Stack	Off-gas train routing	

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TABLE 3.2-IV

SUMMARY OF PROCESSING AND NUCLEAR CONTROL FOR CAN REMOVAL WASTE PROCESSING

Item	Control Limit	Control Method	Comments
Can removal waste NaOH content	≥5% NaOH	Chemical additions	
Retention time of can removal waste in plant	≤24 hours	Chronometer	
Centrifuge speed	≥1200 rpm	Rpm meter	
Centrifuge feed rate	10 gpm	E3 drop-out	
Centrifuge caustic wash solution	25% NaOH 70° C	Chemical make-up	
Centrifuge slurry-out solution	50% HNO_3 0.025 $\underline{M}$ F 4 moles Al per mole F	Chemical make-up	
Thoria dissolution digestion	8 hours 70° C	Chronometer, Thermometer	Double batch if necessary
Pu in E1, E2, E3, E4, or E5	≤ 250 gms Pu	Avoidance of Pu additions	
U-233 and Pa-233 g/t	≤2200 gms U and Pa per ton Th	IPD records	

[redacted]

[redacted]

TABLE 3.2-V

DISSOLVER LIFTER-CIRCULATOR OPERATION DURING THORIA DISSOLUTION

Dissolver Operation	Inner Lifters	Outer Lifters
Jetting can removal waste	Steam (300 pph)	Steam (500 pph)
Acid and chemical additions	Steam (300 pph)	Steam (500 pph)
Heating or cooling solution digestion at boiling	Air (125 cfm to 250 cfm)	Steam (500 pph)
Moving thoria from outer annulus back to inner annulus after 6, 9, 12, etc. hours of boiling	Air (125 cfm)	Air (150 cfm for 5 minutes)
Specific gravity reading after 10, 12, 14, etc. hours of boiling	Steam (300 pph)	Steam (500 pph)
Jetting thorium solution	Steam (300 pph)	Steam (500 pph)
Rinse	Steam (300 pph)	Steam (500 pph)

[redacted]

[redacted]

TABLE 3.2-VI

SUMMARY OF PROCESSING AND NUCLEAR CONTROL FOR THORIA DISSOLUTION

Item	Control Limit	Control Method	Comments
Dissolvent	60% HNO_3 0.025 $\underline{M}$ F 4 moles Al/mole F	Sample and analysis, chemical additions	
HNO_3 required	10 moles HNO_3/mole ThO_2	Charge size, chemical addition	
Dissolver heel	$\leq$ 800 lbs ThO_2	Charge size, previous heel size, thoria dissolved	
Off-gas gamma monitors			Monitors should be adjusted to read both I-131 and Ru-103,106
Off-gas routing	Through back-up facility	Off-gas train routing	
Dissolver rinse	300 gal demineralized water	Wt. Factor	

[redacted]

[redacted]

TABLE 3.2-VII

SUMMARY OF PROCESSING AND NUCLEAR CONTROL FOR ACID BOIL-OFF

Item	Control Limit	Control Method	Comments
Thorium concentration after concentrating solution	4 M Th	Throium input, solution volume, boiling temperature	
Nitric acid concentration at end of acid boil-off	0.5 M to -0.5 M HNO_3 before dilution (b.p. 124° C)	Boiling point temperature	Initially sampling will be necessary
Thorium concentration after dilution	1.5 M Th	Thorium input, solution volume,	
Off-gas monitor			Monitors should be adjused to read both I-131 and Ru-103,106
Off-gas routing	Through back-up facility	Off-gas train routing	
U-233 and Pa-233 g/t in D1, C3 or D5	2200 gms U and Pa per ton Th	Irradiation history, sample and analyses	
Pu in D1, C3 or D5	$\leq$250 gms Pu	Administrative Control of Pu additions, sample and analyses	

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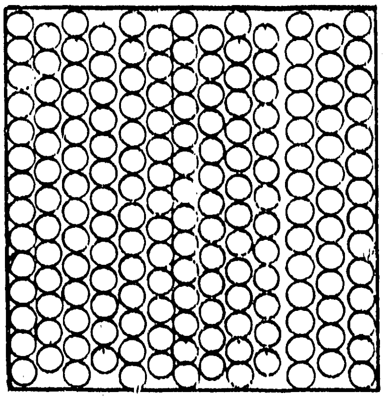
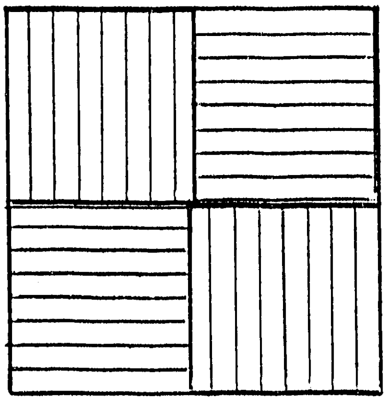
[redacted]

FIGURE 3.2-I

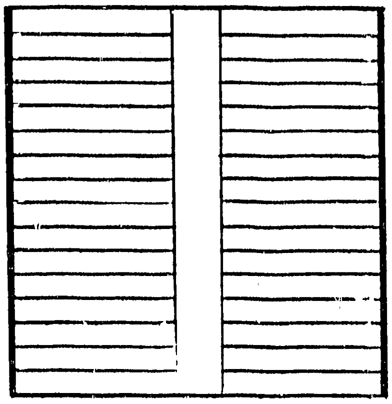
SLUG BUCKET STACKING ARRANGEMENTS



1. Thoria Target Elements



2. Purex Natural Uranium Elements



3. Redox Enriched Uranium Elements

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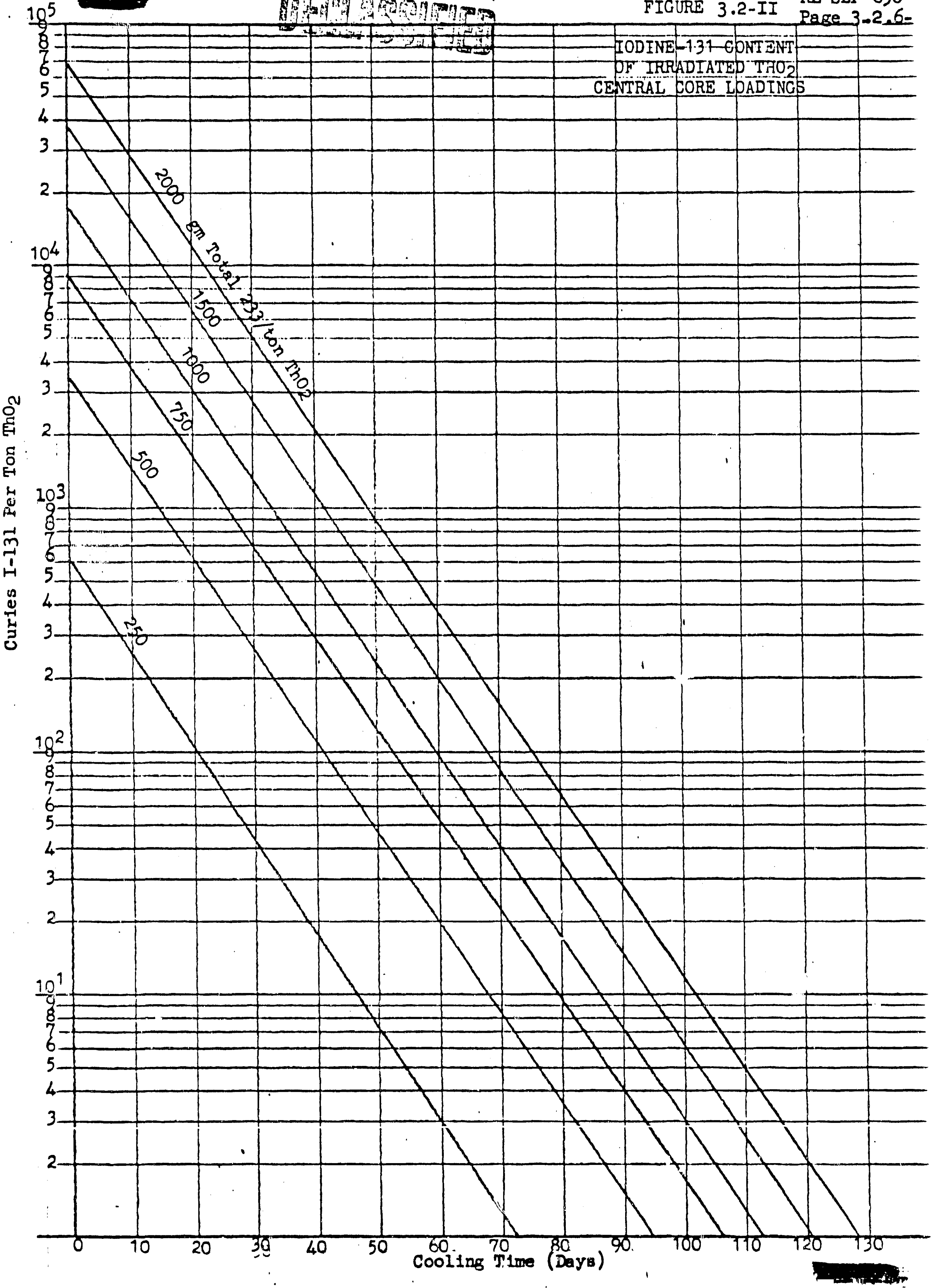
FIGURE 3.2-II



IODINE-131 CONTENT OF IRRADIATED THO$_2$ CENTRAL CORE LOADINGS

[redacted]

[redacted]

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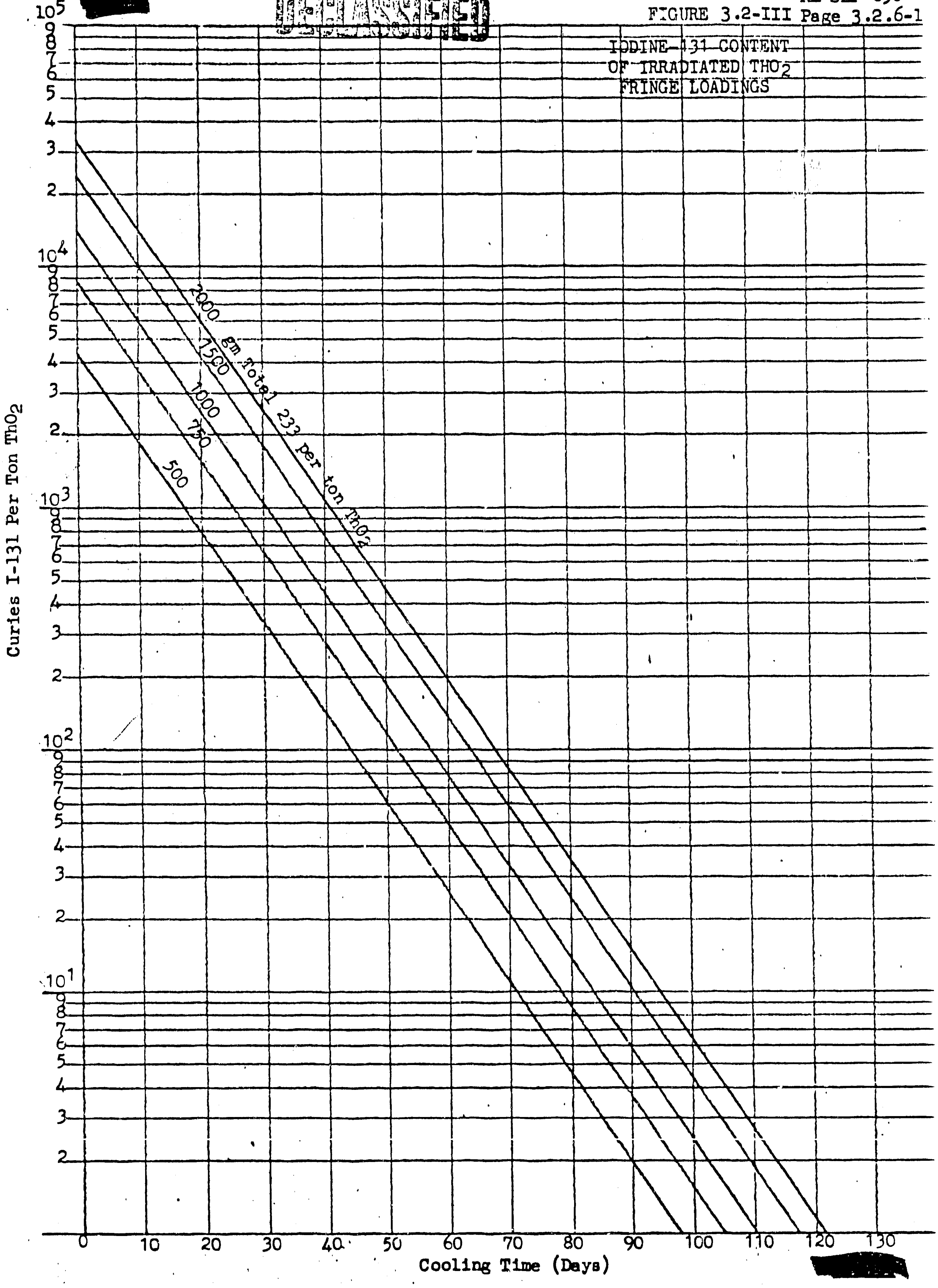

IODINE-131 CONTENT OF IRRADIATED ThO_2 FRINGE LOADINGS

[redacted]

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[redacted]

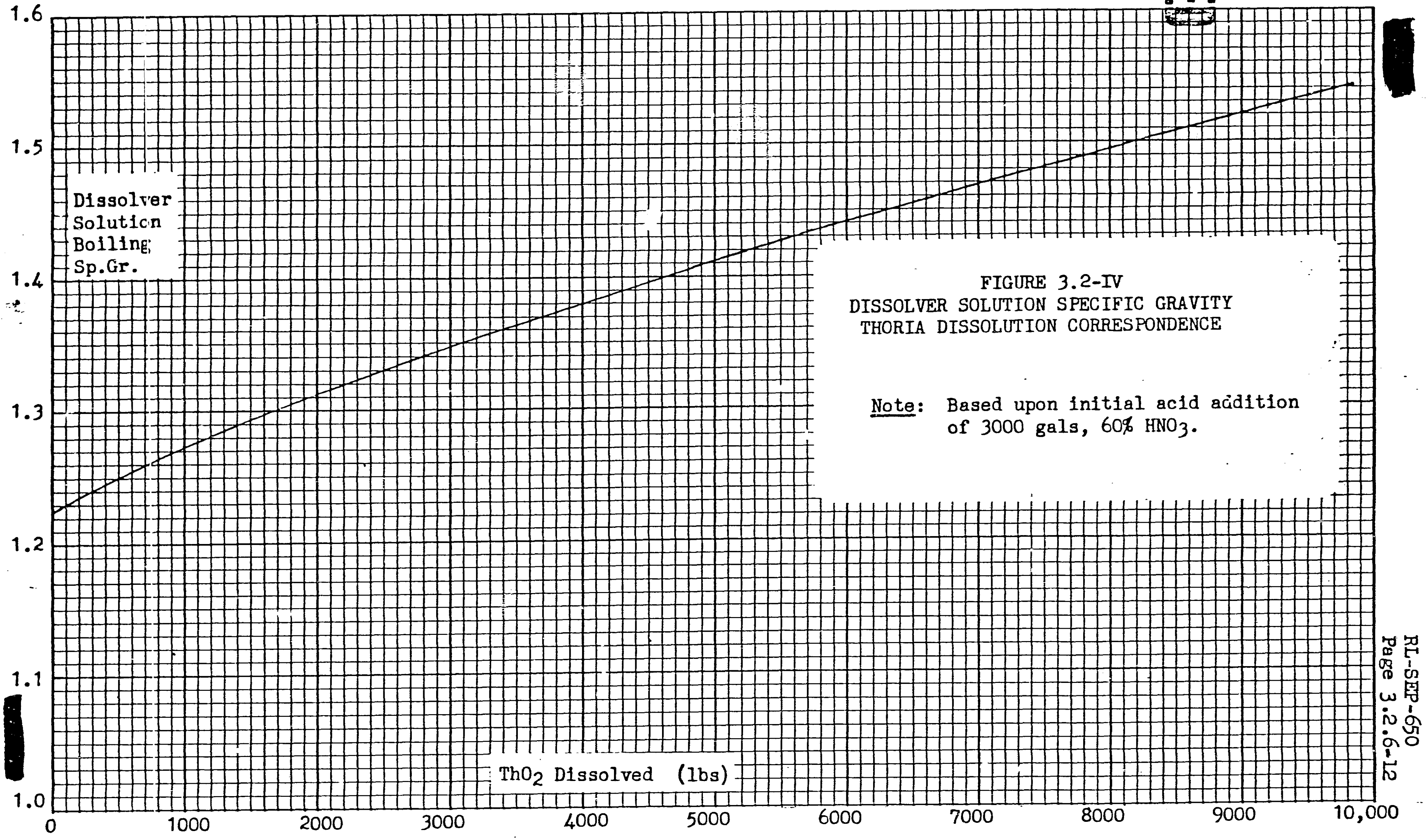

FIGURE 3.2-IV
DISSOLVER SOLUTION SPECIFIC GRAVITY
THORIA DISSOLUTION CORRESPONDENCE

Note: Based upon initial acid addition of 3000 gals, 60% HNO_3.

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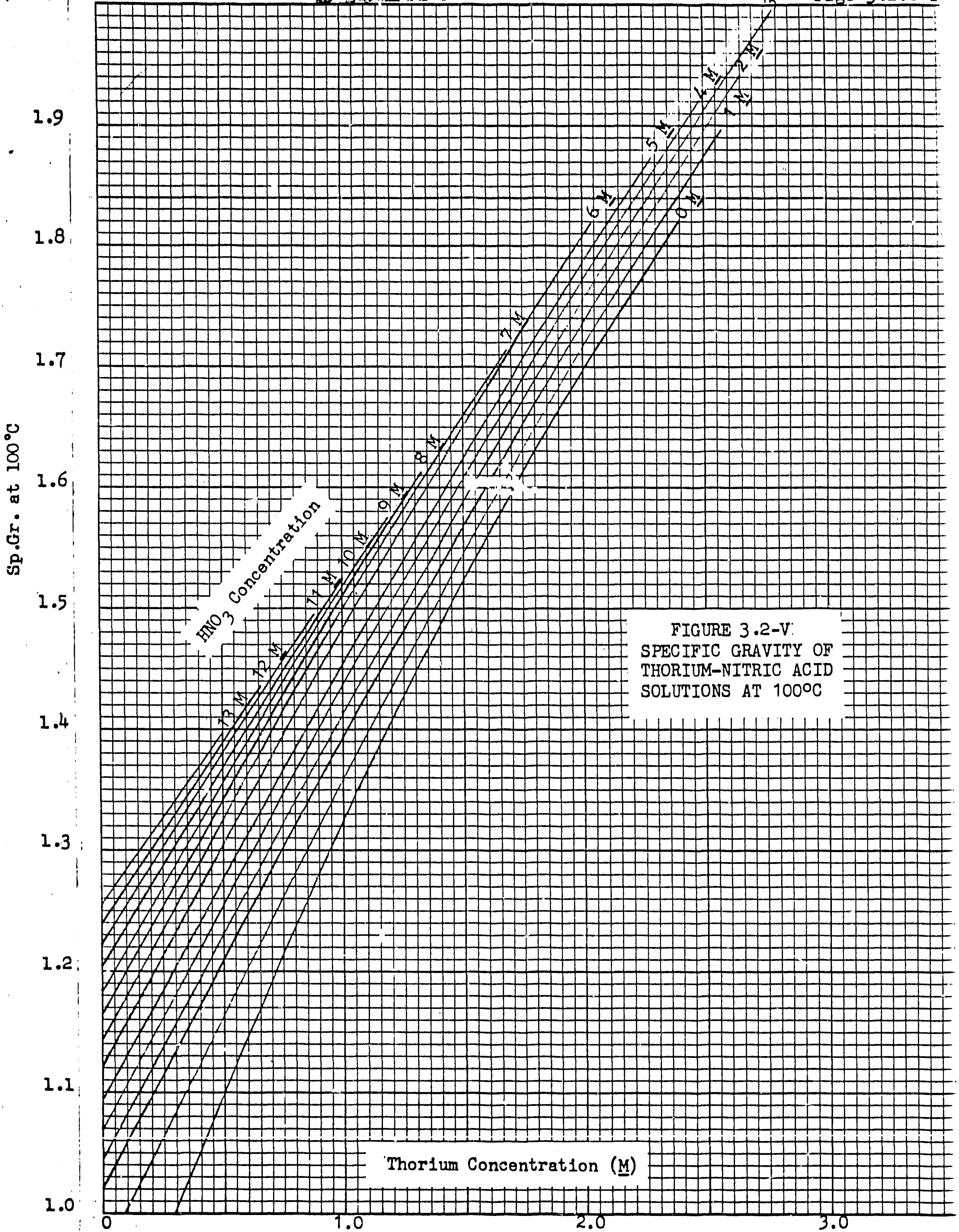

FIGURE 3.2-V
SPECIFIC GRAVITY OF
THORIUM-NITRIC ACID
SOLUTIONS AT 100°C

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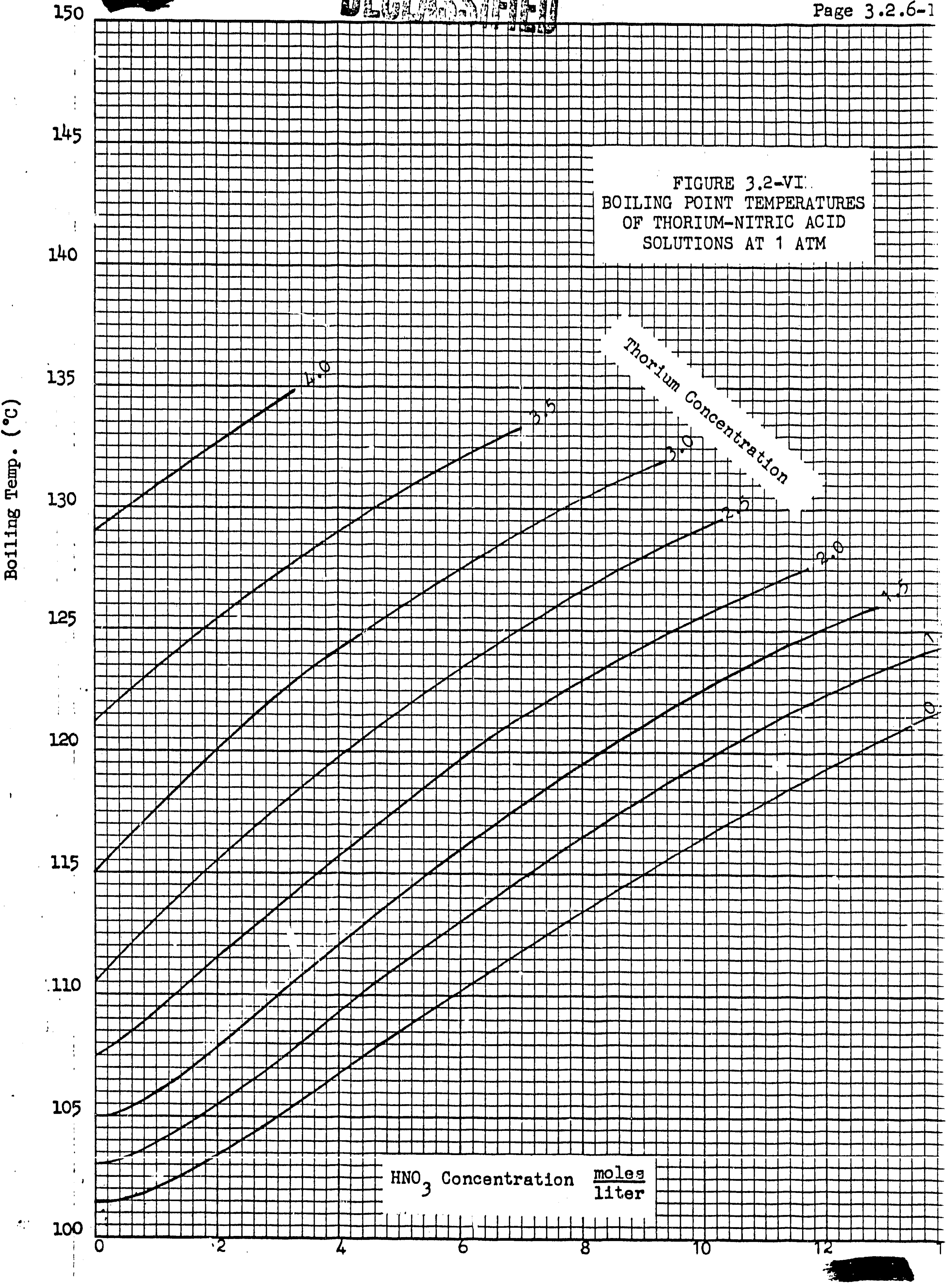

FIGURE 3.2-VI
BOILING POINT TEMPERATURES
OF THORIUM-NITRIC ACID
SOLUTIONS AT 1 ATM

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3.3 SOLVENT EXTRACTION

3.3.1 General

The primary solvent-extraction process control considerations for thorium-uranium -233 processing, as compared to uranium-plutonium processing, are summarized below:

1) Criticality control is primarily concerned with uranium-233, rather than plutonium.
2) The separation factor between uranium and thorium is considerably less than between uranium and trivalent plutonium. Hence process control for partitioning must be more exact.
3) The second the third cycle aqueous waste streams are not concentrated and backcycled. Therefore, process control to minimize losses from these streams is more demanding.
4) Concentration control is required in some processing equipment.
5) The continuous alpha monitors are considerably less sensitive.

The above considerations and their effect on operation and control of the Solvent-Extraction System, in general, are discussed in the following paragraphs.

The general philosophy of criticality control and its application to routine processing in the Purex Plant, as discussed by Judson and Oberg,(1) also apply to thorium-uranium-233 processing. The major differences (and potential problem areas) are: a) the total fissile material content (Pu, U-233, Pa-233) must be considered; b) the safe geometry for concentrated U-233 solutions is less than for concentrated plutonium solutions; and c) the equilibrium organic-phase concentration is approximately two-fold, higher for uranium than for plutonium at corresponding aqueous-phase solute and salting agent concentrations. The potential concentration of

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(1) Judson, B.G. and G. C. Oberg, <u>Process Specifications for Critical Mass Control-Purex Plant (Rev. No. 3)</u>, HW-71625. October 15, 1962, (Secret).

[redacted]

[redacted]

uranium by extraction in a TBP phase requires strict controls on the concentration of uranium and salting agent in any aqueous stream that may come in contact with process solvent.

Personnel must be familiar with the idea of criticality control in connection with uranium, as well as plutonium. For simplicity and to insure being "on the safe side", total alpha count and total uranium analyses should always be considered as 100 percent uranium-233 for criticality control purposes.

The separation factor between uranium and thorium is at least tenfold less than between uranium and plutonium III. To achieve satisfactory separation between uranium and thorium therefore, requires much closer control of the flow ratios and acidities in the partition system.

Since the aqueous waste streams from the second and third decontamination cycles are not recycled, close control is required to maintain losses at low levels. Moreover, the necessity to achieve additional decontamination between uranium and thorium in the second and third decontamination cycles, coupled with their relatively low separation factor requires very close control of the flow ratios and acidities.

Many of the methods and instruments used for process control during normal uranium-plutonium separations are of questionable usefulness for thorium-uranium-233 processing. For instance, the uranium photometer on the 2DW stream, the IBXP neutron monitor, and the H3 gamma monitor cannot be used. The E6 neutron monitor and L1 alpha

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monitor will be useful, but will not be nearly as sensitive because uranium-233 emits about eight-fold fewer alpha counts per unit mass than does plutonium-239. The L1 alpha monitor will be required to measure some 200-fold fewer counts per minute during uranium-233 processing because of the fewer counts per unit mass combined with much lower 3AW waste losses. Consequently, the L1 monitor is useful only as a first indication of intolerable losses. The gamma monitors will be useful in indicating trends in decontamination performance. Other instrument readings, particularly the HA and 2D Column differential pressure measurements, will be useful in detecting column instability. However, the distribution coefficient (E_a^o) for thorium is much less than for uranium in 30 percent TBP-aqueous nitric acid systems. Because of the low solvent saturation the HA Extraction D.P. will probably be insensitive to solvent saturation.

In summary, most instrument measurements will be different than for normal uranium processing and, consequently, considerable data should be analyzed and the instrument measurements correlated before they are relied on for process control.

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3.3.2 Co-Decontamination and Partition Cycle

General operation (including startup) and control of the HA Column is essentially the same as for normal uranium processing. On the initial startup with irradiated thorium, the suggested procedure is to bring the entire solvent-extraction system to steady-state operation under flowsheet conditions, using a dilute sodium nitrate solution in place of a concentrated thorium nitrate solution for the HAF feed stream. The transition to hot feed should be gradual, by adding TNT feed to a relatively full H1 Tank. All streams should be maintained at flowsheet rates until sample analyses indicate a change to be in order.

As discussed in Section 3.3.1 above, the solute (thorium) concentration in the organic phase is much lower than in normal uranium processing. Therefore, the extraction DP is not particularly sensitive to HA Column waste loss.

The primary concern is criticality control, with waste losses of secondary concern. All samples which provide an indication of the uranium-233 inventory (specially designated on the sample schedule) should therefore be expedited. Also, to help in criticality control as well as process control in general, a continuous material balance should be kept on the uranium-233. The accumulation of U-233 in critical vessels (such as T-J6, T-J7, T-H3, E-H4, TK-J1, and TK-J21) is maintained at safe levels by close control of the compositions, temperatures, and flow rates of processing streams. Specifically, (1) the 1BXF uranium concentration is maintained at less than 0.45 g/gal, (2) the 1BX nitric acid concentration is maintained greater than 0.12 molar, (3) the 1BX/1BXF flow ratio is maintained less than 0.70, (4) the 1BX Column temperature is maintained

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less than 35° C, and (5) the solvent is maintained between 28 and 32 volume per cent TBP.

Since critical mass control in the 1BX Column is dependent primarily on control of the 1BX acidity, 1BX Column temperature, and aqueous-to-organic flow ratio, shutdowns and start-ups of the 1BX Column must be made in a manner that will fulfill the critical mass control criteria. In order to meet these criteria, it is necessary to maintain the 1BX-H_2O, 1BX-HNO_3 and 1BXF flows at ratios that will always fulfill the critical mass specifications with respect to the 1BX acidity and 1BX Column aqueous-to-organic flow ratio. An additional safeguard for criticality control involves "stripping" the 1BX Column contents free of uranium prior to shutdown whenever possible. This is most easily accomplished by jetting the 1BSU, and additional fresh organic, from TK-J2 to TK-J3 and processing it through the 1BX Column (after shutdown of the HA Column) until the 1BU sample analysis is ≤ 0.1 g/gal.

Satisfactory removal of the thorium from the 1BU stream requires that (1) the 1BX Column temperature be greater than 25° C, (2) the aqueous-to-organic be greater than 0.5, (3) the 1BX acidity be less than 0.2 $\underline{M}$ HNO_3, and (4) the volume per cent TBP be less than 32.

Control of the 1BX acidity and flow rate within the specified limits is facilitated by high and low loading-pressure alarms on the 1BX-H_2O and 1BX-HNO_3 streams.

Criticality control in the 1BS Column (T-J7) and HAO Tank (TK-J2) is accomplished by adjustment of the aqueous-to-organic flow ratios in the 1BS and 1BX Columns so that the 1BSU-HAO uranium concentration (measured

[redacted]

in TK-J2) does not exceed 9.1 g/gal. The quantity of U-233 in the 1BXT stream can also be substantially altered by changes in the thorium and nitric acid concentrations of the HAP. In addition, the volume of organic in TK-J2 should be controlled so that the mass limit of 3 Kgs of uranium is not exceeded.

The first point at which a continuous indication of decontamination performance is obtained is the 1BT Concentrator (J8 Beckman). The activity of the concentrated thorium stream should be about the same as the 1UC during normal uranium processing.

Criticality control of the 1C Column (T-H3) and 1CU Concentrator (E-H4) is maintained by controlling the aqueous-to-organic flow ratio greater than 0.45 in the 1C Column and maintaining the required 1UC overflow rate. The uranium concentration of the 1UC solution is too low for the specific gravity to be of any use in controlling the boil-off in E-H4. It is therefore necessary to control the uranium concentration in E-H4 by controlling the rate of overflow to the 1UC Receiver (TK-J1). This can be accomplished by maintaining a constant level in TK-J1 and a known flow rate from TK-J1 to TK-J21 to T-J22 (by means of TK-J1 instruments and the J1-6 and 2AF flow control systems, respectively). It must be kept in mind too, that the maximum permissible concentration of uranium in the aqueous phase in the 1CU Concentrator and 1UC Receiver (TK-J1) is a function of the total nitrate concentration. Therefore, if the 1CU nitric acid concentration should be increased (such as by a higher 1BX or 1CX acidity), the 1UC overflow rate should be increased so that the 1UC uranium concentration is maintained within the safe region shown in Figure 4.4-I. The primary cause for concern regarding this portion

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of the process is the possibility of flooding in the 1C Column (T-H3) which would cause gross quantities of solvent to be introduced to the concentrator. The diluent would be stripped (or boiled) off, leaving a 100 per cent TBP organic phase. This concentrated organic phase in turn could extract a large quantity of uranium from the aqueous phase.

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3.3.3 Second Uranium Cycle

The operating and process performance of the Second Uranium Cycle is expected to be very similar to that of the Final Plutonium Cycle during normal uranium-plutonium processing. Therefore, the process and operational control practices, including startups and shutdowns, should be very similar to those used for the Final Plutonium Cycle. It should be remembered, however, that the 2AW stream is not backcycled during thorium processing. Moreover, there is no in-line monitor to measure the 2AW (T-J22) uranium losses and therefore all streams must be maintained at the desired flows and compositions for optimum performance.

The most significant process control aspect of the Second Uranium-233 Cycle is in the area of criticality control. While the methods and philosophy are the same as for normal uranium processing, the limits are generally more restrictive. The individual vessels and columns in the J-Cell Package (except the HAO Tank, TK-J2) are safe for U-233 solutions of 40 g/l. However, because of the unsafe J-Cell sumps, the criticality control limits must be set at 5.5 g/l for Tanks J1 and J21 and 8 g/l for the T-J22 and T-J23 columns. This limit is in turn met by controlling the 2AF uranium and nitrate concentrations, the 2AS acidity, and the aqueous-to-organic flow ratios in the 2A and 2B Columns.

The possibility exists, and it therefore must be assumed, that a solvent phase may be present in any vessel in the Solvent Extraction System. Moreover, the uranium concentration in the solvent phase can be considerably higher than in the aqueous phase. This is especially true in the case of the 2AF Tank (TK-J21) since 100 per cent TBP could be

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formed in the 1CU Concentrator, overflowed to the 1UC Receiver (TK-J1), and be pumped to TK-J21. The practical criticality control limit in the Second Uranium Cycle is therefore that aqueous-phase uranium concentration which is in equilibrium with an organic-phase (100 per cent TBP) uranium concentration of 5.5 g/l. As shown in Figure 4.4-I, the maximum permissible aqueous-phase uranium concentration is a function of the salting strength, reaching an upper limit of 0.95 g/l at the corresponding 1UC nitrate concentration of 0.36 molar. Because of this relationship, a restriction is also required on the total nitrate concentration in the 2AF in the event an organic phase is present in either TK-J1 or TK-J21. In order to minimize the HNO_3 in the 2AF (J-21) Tank, the 2AF-HNO_3 stream is routed directly to the 2A Column (T-J22) via the middle differential pressure tap.

The 2BU stream will overflow back to the 2AF (J-21) Tank if the 2BU jet fails to function. This recycle could cause the criticality control limits for TK-J21 to be exceeded and thus lead to loss of criticality control and possibly excessive 2AW uranium losses.

Additional requirements to insure maintaining the uranium concentration within the criticality control limit of 8 g/l in the Second Uranium Cycle columns include the following limits on the feed-to-extractant flow ratios in the 2A and 2B Columns (T-J22 and T-J23):

2AF/2AX - 5.0, Max.
2BX/2AX - 0.6, Min.

Control of the stream flow rates within these limits is facilitated by a high loading-pressure alarm on the 2AF flow control valve and low loading pressure alarms on the 2AX and 2BU flow control valves.

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3.3.4 Third Uranium Cycle

Process control methods for the Third Uranium Cycle are not significantly different from current practices. Since differences result, however, from (1) critical mass consideration, (2) no 3BW sampler, and (3) no 3AW backcycle, these differences in general permit less latitude with respect to process conditions.

Critical mass control of the system requires monitoring of the nitrate and uranium concentration in TK-J5 and TK-N1 to assure critical mass control in the event a 30% TBP phase is present in either of these tanks. In order to minimize the acid concentration in TK-J5, the 3AF salting agent is added directly to the 3A Column (T-L1) via the middle dp line. Since the T-L1 differential pressure instruments are removed from service, the static pressure must be relied upon for column control.

Maintaining the uranium concentration within the criticality control limit of 30 g/l in the 3A (T-L1) and 3B (T-L2) Columns is insured by the following required limits on the respective flow rates:

3AF/3AX - 5.0 max.
3BX/3AX - 0.7 min.

Additional aid in criticality control in the Third Uranium Cycle is provided by the following alarms:

3AF	High valve loading pressure
3AF-HNO_3	High valve loading pressure
3AX	Low valve loading pressure
3BU	Low valve loading pressure
3BU-α Monitor	High alarm
TK-N1	High Wt. Fac. alarm
TK-N1	Low Sp. Gr. alarm
E-N6	Low Wt. Fac. alarm
E-N6	High Sp. Gr. alarm

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Although the sensitivities of the 3AW and 3BU alpha monitors are about 8-fold lower for U-233 than for Pu-239, these instruments are still valuable for detecting 3AW losses and changes in U-233 flow through the plant.

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3.3.5 Second Thorium Cycle

The process control techniques for the Second Thorium Cycle are essentially the same as during normal uranium processing. However, since thorium is more difficult to extract and decontaminate than uranium, deviation from flowsheet conditions must be smaller during thorium processing. Satisfactory performance of the Second Thorium Cycle is dependent on many factors, but the 2DF acidity, 2DX acidity, 2D Column extraction L/V, the 2E Column L/V, and the 2D and 2E Column operating temperatures are the most important considerations.

Thorium solutions are colorless. For this reason the 2DW photometer cannot be used to monitor 2DW thorium losses. Except for the 2DW photometer, all of the remaining instrumentation for the Second Thorium Cycle should be useful for instantaneous process control.

A 2DW thorium loss between 0.05 and 0.2 per cent will probably be required for satisfactory fission product and plutonium decontamination in the 2D Column. The 2DW thorium losses should be controlled by adjustments to the 2OS-HNO_3 or 2DX flows. Adjusting the 2OS-HNO_3 flow is preferable since the effect on decontamination performance is not great. The fission product decontamination performance of the 2D Column is markedly influenced by the 2DF acidity. Reduction of the 2DF acidity between -0.1 to -0.2 M HNO_3 by the addition of sodium hydroxide to TK-K1 may be required for satisfactory decontamination from ZrNb-95 and Pa-233.

Decontamination of the thorium product from uranium or reflux of uranium-233 in the 2E Column requires close control of the 2E Column L/V and temperature. The 2E Column L/V should be maintained at the

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minimum value that is consistent with a satisfactory 2EW thorium loss (≤ 0.02%). Processing experience may show that the control required to meet flowsheet objectives is impractical, and it may be necessary to periodically purge the uranium reflux from the 2E Column.

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3.4 Final U-233 Decontamination and Concentration

The Third Uranium Cycle product, 3BU, is passed through two columns prior to concentration and loadout. A small lead-shielded column of unfired Vycor glass adsorbs protactinium and the zirconium-niobium fission products from the solution. A larger unshielded column of Dowex-50 cation resin (acid form) absorbs essentially all of the thorium and passes most of the uranium. Most of the uranium which remains on the cation bed during normal operation is displaced by thorium. The residual uranium is eluted with stronger nitric acid after the cation bed is saturated with thorium. Uranium elution is required when 750 grams of thorium have been fed to the cation column. Pretreatment of both column beds with nitric acid is necessary. The cation bed must be converted from the sodium form to the acid form, and the Vycor glass bed must have the boron leached out prior to U_{233} processing.

Normal operation of the columns consists of passing 3BU-3UF solution from TK-N1 through the Vycor and cation columns, then back to the 3UC Concentrator, E-N6. Periodic radiation monitoring of the columns is necessary. If the radiation approaches objectionable levels, the 3UF solution bypasses the columns and the cation column is eluted with 0.5 M HNO_3 to TK-J1 in order to recover the uranium product. The thorium and fission products are eluted later and routed to TK-F10 as waste. If radiation from the fission product adsorption column approaches an objectionable level, the column can be removed.

A flow sketch is shown in Figure 3.4-I.

[redacted]

TABLE 3.4-I

N-CELL FLOW SKETCH

U-233 PROCESSING

LOAD-OUT HOOD VALVES

1. Cation Column Effluent to E-N6
2. Columns Drain to Load-Out Hood Floor
6. Adsorption Column Effluent to Cation Column
7. Adsorption Column Feed from TK-N1
8. Adsorption Column By-Pass, TK-N1 to Cation
9. Columns By-Pass, TK-N1 to E-N6
11. Adsorption Column XCX Feed
12. Cation Column XCX Feed
14. Load-Out Tank Vent
15. Load-Out Tank Drain (Double) to Load-Out Bottle
16. Load-Out Head Tank Feed from TK-N7
17. Load-Out Head Tank Drain (Double)
31. Columns Feed from TK-N1
32. Load-Out Hood XCX Flush
33. Columns XCX to XAW Jet

3 Way Load-Out Head Tank, Vacuum or Vent

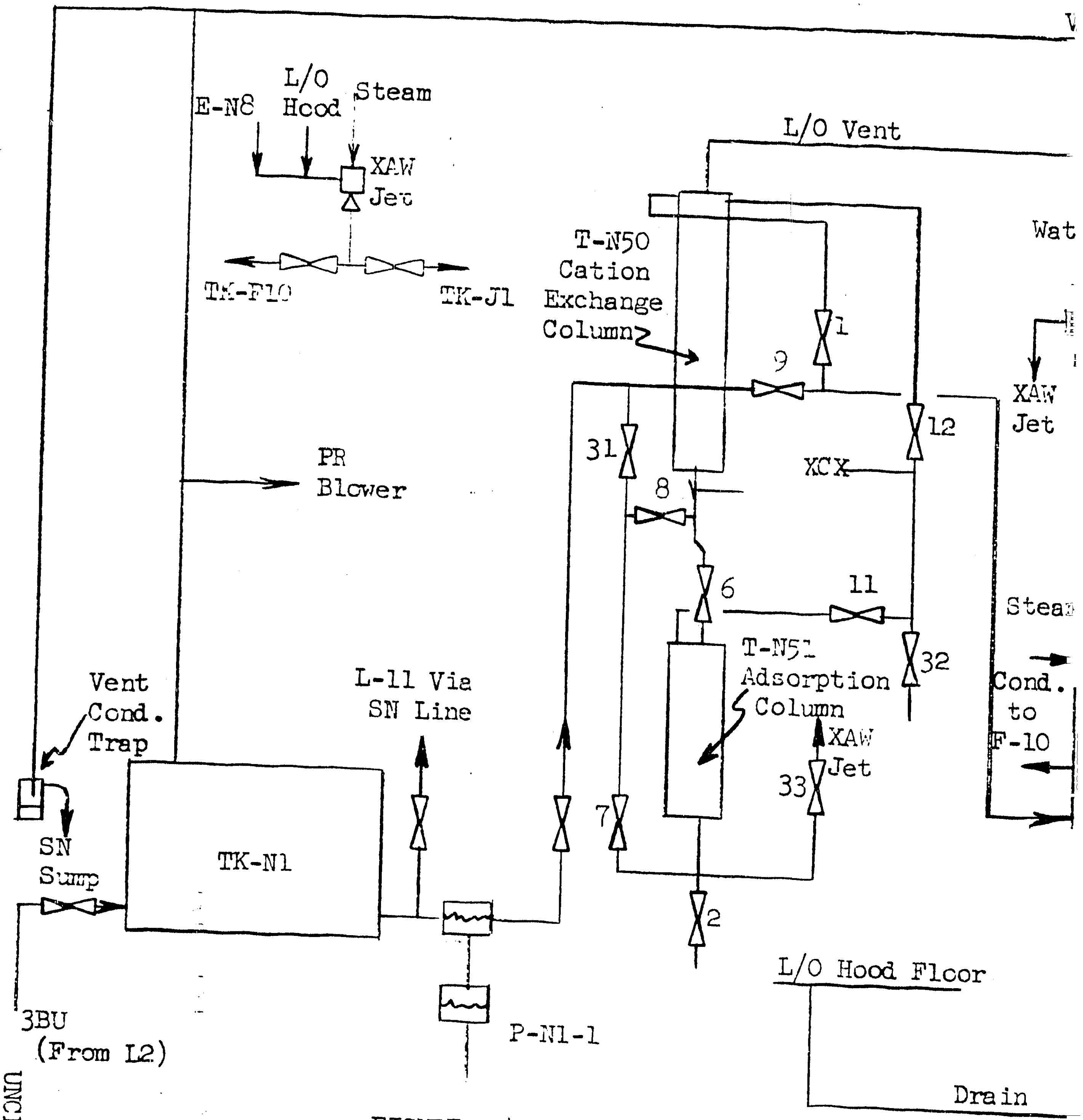

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FIGURE 3.4-I

N-CELL FLOW SKETCH

U-233 PROCESSING

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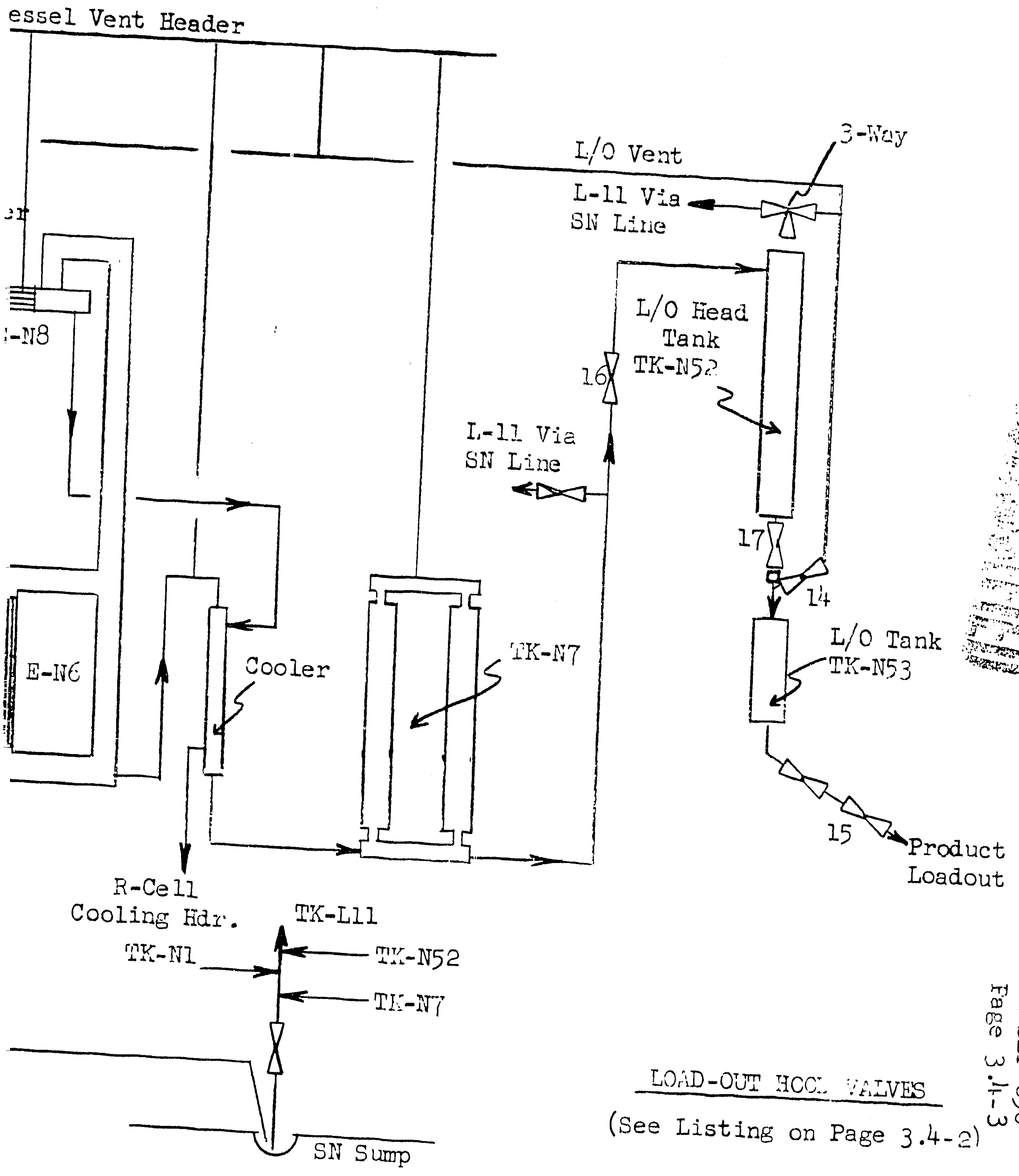

LOAD-OUT HCCL VALVES

(See Listing on Page 3.4-2)

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3.5 Waste Treatment And Acid Recovery

Operation of the Waste Concentration and Acid Recovery Systems during the thorim run will be similar to normal uranium processing operation. The following lists several exceptions to normal operations and points of special interest with respect to waste concentration and acid recovery:

(1) Waste streams to be concentrated in EF6 will be routed from the Codecontamination Cycle (HAW), Second Uranium Cycle (2AW), Third Uranium Cycle (3AW) and the Second Thorium Cycle (2DW). The 2AW, 3AW and 2DW are batch transferred from Tank F10 to Tank F12.

(2) The 1WW overflow from E-F6 to TK-F7 will be about 1.1 times normal. This is necessary to keep the total aluminum and sodium concentrations at a level that will prevent precipitation.

(3) Boiloff from the thorium denitration step (From C3 Dissolver) will be routed to Tank F3. If, during the run, it is found that this acid is too radioactive to add directly to TK-F3 it will be added to TK-F12.

(4) If the A3 and B3 dissolver towers cannot be sufficiently decontaminated from uranium, it will be necessary to route the reflux acid to Tank F12 for acid recovery.

(5) The aluminum nitrate used for complexing fluoride in the dissolvers will be added batchwise to the AAA stream (TK-F3) just prior to transfer to U Cell for acid recovery. The amount of aluminum to be added will be calculated to give an aluminum concentration of 0.05 $\underline{M}$ in the Purex Recovered Acid.

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(6) The U6 Fractionator will be operated to produce 60 per cent Purex Recovered Acid for use in the dissolver; otherwise, operation will be as usual.

The 1WW flow during the thorium run will be about 10 per cent higher than for normal uranium plutonium production, thus requiring a greater load on the sugar denitration system. The plan is to utilize Tank F15 for sugar denitration and Tank F16 for digesting and neutralization.

If it is necessary to route either the A3 and B3 reflux or the C3 denitration acid to TK-F12, it will be impossible to keep up with sugar denitration and the acid will have to be neutralized and sent to UGS. The following paragraph outlines a method of handling the large quantities of acid if C3 overheads are routed to TK-F12.

(1) Transfer the batch (~3600 gallons) of 1WW from TK-F7 to TK-F15.
(2) Heat to operating temperature and begin adding sugar.
(3) Add sugar for ~20 hours at maximum rate (do not over-sugar based on TK-F7 acid analyses).
(4) Cool and transfer to TK-F16.
(5) Transfer a new batch to TK-F15 and begin again.
(6) Digest sugar treated waste in Tank F16 for 4-8 hours, cool and sample.
(7) Neutralize, sample and send to UGS.

During the neutralization step certain precautions should be taken:

(1) The aluminum concentration of the 1WW should be less than 0.5 M. (Above 0.5 M Al, it is recommended that "reverse strike" neutralization be used.)

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(2) Add the sodium hydroxide as rapidly as possible and maintain TK-F16 temperature below 100° C. It is desirable to hold the temperature above 50° C during neutralization to aid in preventing aluminum precipitation.

The neutralized waste is to be stored in nonboiling waste storage tanks. It is necessary to have a final pH of at least 12 to protect the waste routings and to guard against nitric oxide release to the atmosphere.

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3.6 Solvent Treatment

The solvent treatment systems will be operated much the same as during regular uranium processing with the following exceptions:

(1) Transfers from Tank R1 to Tank G1 or from Tank R2 to Tank G2 will be eliminated in order to avoid possible cross-contamination by solvent degradation products.

(2) The 10B $KMnO_4$ stream to Tank G1 will be eliminated since ZrNb decontamination will not be a problem.

(3) The 20S HNO_3 rate will be higher than normal, to insure the required acidity in the 20S stream.

(4) Changeout frequencies and volumes will be as follows:

- (a) TK G1 ---- 600 gallons every 8 hours
- (b) TK G2 ---- 600 gallons every 24 hours
- (c) TK R1 ---- 600 gallons every 24 hours
- (d) TK R2 - Initially add 300 gallons acid make-up; then after 24 hours operation, empty out $\sim$900 gallons. (Buildup is due to additional 20S HNO_3 flow.)

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3.7 Sample Schedule

The sample schedule recommended during continuous steady-state thorium processing is shown below. The requirements listed in this sample schedule are considered the minimum analyses and frequency that can be tolerated with respect to the specification in Section 4.0. During plant start-up, plant shutdown, and abnormal processing situations, additional samples and analyses are of course needed for satisfactory process control.

Feed Preparation

Stream	Sample	Sample Frequency	Analyses
Dissolver TNT	A3 and B3	Batch (total of two per day until satisfactory controls are established.)	HNO_3 AT Gamma Scan (Pa, Zr-Nb, Ru) Th
Stock TNT	C3	Batch (total of one per day until satisfactory controls are established.)	HNO_3 Th AT
Stock TNT	D5	Batch (one per day)	Gamma Scan HNO_3 Th AT U-233 Pu Total Uranium
		3 Day Composite	U Isotopes Fe F Al
Centrifuge Recycle	E1	Batch (one per day)	HNO_3 AT Th Gamma Scan (Pa, Zr-Nb, Ru)
Centrifuge Waste	E5	Batch (two per day)	pH Th AT Gamma Scan (Pa, Zr-Nb, Ru)

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Stream	Sample	Sample Frequency	Analyses
HAF	E6*	Batch (one per day)	HNO_3 Th } Batches AT } Containing Pu } Rework

Co-Decontamination and Partition Cycle

Stream	Sample	Sample Frequency	Analyses
HAW	H2	One per shift	Th HNO_3
1BXF	J3*	One per shift One per day	AT Gamma Scan
1BSU	J2*	One per shift	AT
1BU	J7*	One per shift	AT Th
1BT	J8*	One per shift	AT HNO_3 Th Visual
1CX	1CX	One per day	HNO_3
1BX	1BX	One per shift	HNO_3
1CW	H3*	One per shift	AT

Second Uranium Cycle

Stream	Sample	Sample Frequency	Analyses
1UC	J1*	Two per shift	HNO_3 AT
		One per day	Th Gamma Scan
1UC	J-21*	One per shift	HNO_3 AT
		Five-day composite	Uranium Isotopes
2AW	J-22*	One per shift	AT HNO_3
2BW	J-23*	One per shift	AT

* Samples used to measure U-233 inventory for criticality control.

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Third Uranium Cycle

Stream	Sample	Sample Frequency	Analyses
3AF	J5-B*	One per shift	HNO_3 AT Gamma Scan
		One per day	Th
3AW	L1*	One per shift	AT HNO_3
3BU	L3*	One per shift	HNO_3 AT Th Gamma Scan
3BW	L2*	One per day	AT
3UC	N7*	Batch	U-233 HNO_3 Gamma Scan AT Th Fe SpG
		Five-batch Composite	Pu SO_4 Cl TMI Uranium Isotopes

Second Thorium Cycle

Stream	Sample	Sample Frequency	Analyses
2DF	K1*	One per shift	AT HNO_3 Th
		One per day	Gamma Scan
2DW	K2	One per shift	HNO_3 Th
2EW	K3	One per shift	Th AT
2ET	K4	One per shift	Th Gamma Scan AT Visual

* Samples used to measure U-233 inventory for criticality control.

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Second Thorium Cycle (Cont'd)

Stream	Sample	Sample Frequency	Analyses
TNT Product	K6	Each batch	Th HNO_3 U-233 Total Uranium Gamma Scan AT Pu SpG
		Five-Batch Composite	TMI SO_4 Cl UX_1
2DIS	2DIS	One per day	HNO_3 PO_4

Solvent Recovery

Stream	Sample	Sample Frequency	Analyses
20W	R8	Batch (one per day)	pH Th AT
10W	G8	Batch (one per day)	pH Th AT
10D	G7	One per shift	pH
100	G5	One per day	Gamma Scan (Pa,Zr-Nb,Ru) Pu Ret
		Twice per week	% TBP
200	R7	One per shift	HNO_3
		One per day	Gamma Scan (Pa, Zr-Nb, Ru) Pu Ret
		Twice per week	% TBP

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Waste Concentration and Acid Recovery

Stream	Sample	Sample Frequency	Analyses
AAD	F5	One per day	HNO_3 Gamma Scan Total Beta
AAA	F3	Batch (four per day)	
		Daily Composite	HNO_3 Gamma Scan F Fe Al
AFD	U6 Tail-Water	One per day	$pH(HNO_3\text{-}pH \leq 2)$ Gamma Scan Total Beta
Purex Recovered Acid	U1 and U2	Batch (one per day)	HNO_3 Gamma Scan Al
		3-day composite	F Fe
2WF	F10	One per shift	AT

Sugar Denitration and Neutralization

Stream	Sample	Sample Frequency	Analyses
1WW	F7	Batch (one per day)	HNO_3 Th Al U-233 Pu Total Uranium
STW	F-15 and F-16	Batch (one per day)	Caustic Ratio
STW-NW	F-15 and F-16	Batch (one per day)	pH

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AMU

Stream	Sample	Sample Frequency	Analyses
HAS	TK-209, 210	Each Batch	HNO_3 H_3PO_4 Fe++
2AS	TK-220, 221	Each Batch	HNO_3
2BX	TK-224, 225	Each Batch	Fe HNO_3
3AS	TK-211, 212	Each Batch	HNO_3
3BX	TK-213, 214	Each Batch	HNO_3
$Fe(SA)_2$	TK-101	Each Batch	Fe++
2DIS-PO_4	TK-218		PO_4

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3.8 Product Rework

3.8.1 Uranium-233 Rework

Uranium-233 solutions scheduled for rework - either impure product or SLA, SLD or SN sump material--- will be recycled to the process in much the same manner as plutonium is recycled during uranium-plutonium processing operations. The major differences are as follows:

(1) The maximum permissible U-233 concentration in the feed solution in TK-E6 is higher (2200 g U-233 plus Pa-233 per ton of thorium) but the g/T ratio of the virgin feed is also higher.

(2) The maximum permissible U-233 concentration in the rework solution is 20 per cent lower (240 g U-233/1 vs 300 g Pu/1) because of the more restrictive E-Cell sump limits for U-233.

(3) Water, rather than nitric acid, is used as the motive and dilution fluid for the L11 to E6 transfer jet. (The water is supplied from the XCX Makeup Tank in AMU so no piping changes are involved.)

(4) TK-L9 and TK-L10 cannot be used for recycle of U-233 solutions at concentrations greater than 75 grams per liter.

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3.8.2 Rework of Thorium Product Solutions

Thorium nitrate (TNT) solutions produced during the campaign that require reprocessing will most likely be recycled directly from TK-K6 to TK-E6. It is not planned that any 'P' tank will be flushed free of uranium to accommodate thorium storage. Recycle from TK-K6 to TK-K1 is possible, but would require shutdown of the Co-Decontamination and Partition Cycle. Rework of TNT product solution via TK-E6 and the entire solvent extraction system might be desirable to (a) reduce U-233 levels in the system or (b) to recycle TNT product that is grossly contaminated with impurities. Recycle from TK-K6 will be batchwise and feed adjustment in TK-E6 will be much the same as routine adjustment of dissolver solution.

A large amount of TNT may be produced (especially at the start of the campaign) that exceeds the specification purity limits by too great a margin to be blended with "good" product or that contains excessive U-238 contamination (which should not be re-introduced into the system). It would be advisable to ship this material to the 200-W vault for segregated interim storage. This contaminated TNT solution could be returned to the Purex Plant for reprocessing at the conclusion of the campaign - after the U-233 has been purged from the system.

Depending on the amount of TNT solution involved, the rework flowsheet may be identical to the one shown in Section 2 or it may be adjusted for special considerations (e.g., a dilute feed for a small amount of solution). For example, a special flowsheet will be

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recommended for processing the TNT product solution from the January 1966 thorium test run. This solution is eight-fold high in uranium contamination and greater than ten-fold high in metallic impurities (primarily sodium) and sulfate contamination.. It will thus require special considerations to prevent contaminating the system with uranium. Furthermore, the relatively small quantity (~7000 pounds of thorium) will require a dilute flowsheet.

The TNT product solution from the test run could be reworked by several methods, either prior to or after completion of the thorium campaign. Rework prior to the campaign is preferrable, however, so the tankage will be available for segregated interim storage of any rework material produced during the campaign.

Thorium nitrate solution requiring rework can be processed through both the Co-Decontamination and Partition Cycle and the Second Thorium Cycle or through the Second Thorium Cycle, only --"spinning", if necessary, to achieve the desired purity. Use of the Co-Decontamination and Partition Cycle prior to the campaign introduces the potential hazard of downgrading the U-233 product with U-238 and, for this reason, is not recommended. TNT solution recycled through the Second Thorium Cycle will be decontaminated from uranium in the 2E Column in much the same manner as in the 1BX Column. The residual uranium and thorium in the 2EW (organic waste) stream will be scrubbed out in TK-R1 and discarded to waste.

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Process control during thorium rework operations should be generally similar to regular operation, except that the thorium concentration in the feed stream may be more dilute.

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3.9 Process Blanks and Isolation

During the thorium processing run it will be necessary to maintain blanks in lines that could carry precipitating agents to vessels containing large quantities of fissile material. Certain blanks will also be needed to isolate stored solutions containing U-238 and Pu-239 from process solutions containing U-233 and Pa-233. These precautions are required to insure good criticality control.

In order to maintain good process control during the run, other blanks and controls not related to nuclear safety will be required. Lists covering the two areas of control are listed below:

(1) Process Blanks for Nuclear Safety

Vessel	Nozzle or Line	Action
Tanks D3-D4	DG15 Steam to Jet D4 to D5 DG46 Steam to Jet D3 to D5	Blank in P&O Gallery Blank in P&O Gallery
Tank D5	DG12 Chem Add DG1 Steam to Jet D5 to D3	Blank in P&O Gallery Blank in P&O Gallery
Tank E5	EG17 Steam to Jet E5 to E6	Blank in P&O Gallery
Tank F10	FG118 Power to Pump F10 to H4 or (FG119) (Steam to 3WF Variable Jet)	Take pump out of service at motor control station. (Blank in P&O Gallery)
Tank F13	FG72 Steam to Jet F13 to J3	Blank in P&O Gallery
Tank G1	GG170 Na_2CO_3 Add	Blank $KMnO_4$ and 7003 Hdr. Add to wall nozzle in P&O Gallery.
Tank G5	GG76 G5A to G5	Blank in P&O Gallery
Column T-H3	Nozzles L, A, Q, P, B	Normally blanked.
E-H4 Concentrator	HG17 7007 Hdr. to H4 Nozzles HH, CC, JJ, U, K	Blank in P&O Gallery Normally blanked

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Vessel	Nozzle or Line	Action
Tank J1	JG174 Steam to Jet J1 to F10	Blank in P&O Gallery
	JG175 Steam to Jet J1 to H1	Blank in P&O Gallery
	JG173 Nitric to J1	Blank in P&O Gallery
Tank J2	JG167 Chem Add to J2	Blank in P&O Gallery
	Nozzle AR on J-Cell Package	Normally blanked
	Nozzles P, M, S, DB	Normally blanked
Tank J21	JG140 Cehm Add.	Blank in P&O Gallery
	Nozzles F, J, K	Normally blanked
Column J22	JG146 HNO_3 to 3A Column	Blank in P&O Gallery
Tank J3	JG136 Chem Add to J3	Blank in P&O Gallery
Column T-J4	JG122 Pulse Water to J4	Remove Quick Connect and blank in P&O Gallery
	JG89 Nitric to J4	Blank in Sample Gallery
Tank J5	JG94 $NaNO_2$ Add to J5 (also 1BX to J5)	Blank in P&O Gallery
	JG95 Chem Add to J5	Blank in P&O Gallery
	JG76 Steam to Jet J5 to J1	Lock & tag in Control Rm.
Column T-J6	JG52 Steam to Jet J6 to J4	Blank in P&O Gallery
Tank K6	KG1 Steam to Jet K6 to J1	Blank in P&O Gallery
Column T-L2	LG35 Steam to Jet 2BP to J5	Blank in White Room
	LG23 HNO_3 to L3 Stream	Blank in rotameter in P&O Gallery
T-L3 Stripper and E-L4 Concentrator	LG23 Chem Add. to L3 & L4	Blank in White Room
	LG29 Steam to L3	Blank in White Room
	LG15 Steam to L4	Blank in White Room
Tank Q5	Blank Q5 to J1 Jet	(Normally blanked)
Tank N1	Blank HNO_3 Add to N1 Tank	Blank in N Cell AMU
	Blank Recycle Line to N1 Tank	Blank in N Cell at N1 Tank
	Blank ANN Add to N1 Tank	Blank in N Cell AMU
Tank N7	Blank N7 to L9/L10 Tanks	Blank in N Cell Hood
Tank 105	Wash Header Lines from 105 Tank to all AMU Tanks listed	Blank line to tanks listed.

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Vessel	Nozzle or Line	Action
	213	
	214	
	215	
	216	
	217	
	218	
	219	
	220	
	221	
	222	
	224	
	225	
	324	
	150	
	151	
	152	
	153	
	103	
	106	
	108	
	204	
	205	
	206	
Tank 108	108 Header Lines to the following tanks: 204 205 206 324	Blank line to tanks listed
	Sodium Hydroxide Addition Lines to the following tanks: 204 205	Blank lines to tanks listed

(2) Process Blanks and Controls for Process Control

Vessel	Nozzle or Line	Action
A3 Dissolver	AG11 Steam to Jet A3 to D1	Lock and tag in H.E. Control Room
	DT34 A3 to E3 or D2	Remove jumper from manifold jumper to Tank D2 and blank.
B3 Dissolver	BG11 Steam to Jet D3 to D1	Lock and tag in H.E. Control Rm.
	DT39 B3 to E3 or D2	Remove jumper from manifold jumper to Tank D2 and blank.

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Vessel	Nozzle or Line	Action
C3 Dissolver	CE3 Steam to Jet C3 to D2	Lock & tag in H.E. Control Rm.
	CG15 Steam Jet C3 to D1	Lock & tag in H.E. Control Rm.
Tank D1	DG59 Steam to Jet D1 to B3	Lock & tag in H.E. Control Rm.
	DG72 Steam to Jet D1 to A3	Lock & tag in H.E. Control Rm.
	DG60 Steam to Jet D1 to E3	Blank in Sample Gallery
Tank D2	DG51 Steam to Jet D2 to UGS	Lock & tag in H.E. Control Rm.
Tank D3-D4	DT23 Alternate D3 to E3	Jet jumper is currently off. If system is restored, lock and tag jet.
Tank D5	DG14 D5 to F8	Lock & tag in H.E. Control Rm. (may be disrupted for A-3-4 to UGS Route)
Tank E1	EG88 Steam to Jet E1 to F18	Lock & tag in H.E. Control Rm.
	EG89 Steam to Jet E1 to UGS	Lock & tag in H.E. Control Rm.
	EG98 Steam to Jet E1 to Load-Out	Lock & tag in H.E. Control Rm.
Tank E3	EG58 Steam to Jet E3 to UGS	Remove jet and blank at ET24
Tank E5	EG22 Steam to Jet E5 to F18	Lock & tag in H.E. Control Rm.
	EG23 Steam to Jet E5 to E3	Lock & tag in H.E. Control Rm.
	EG29 Steam to Jet E5 to F8	Lock & tag in H.E. Control Rm.
Tank E6	EG8 RC Hood to E6	Blank in Sample Gallery
	EG14 Steam to Jet E6 to D5	Lock & tag in H.E. Control Rm.
Tank F3	FG244 Steam to Jet F3 to F10	Lock & tag in Control Rm.
	FG243 Steam to Jet F3 to F16	Lock & tag in Control Rm.
Tank F7	FG167 Steam to Jet F7 to F12	Lock & tag in Control Rm.
	FG160 Steam to Jet F7 to F8	Lock & tag in Control Rm.
Tank F8	FG159 Steam to Jet F8 to E Cell and D5	Lock & tag in H.E. Control Rm.
	FG154 Steam to Jet F8 to F16 (Alternate)	Lock & tag in H.E. Control Rm.
Tank F10	FG133 Chem Add ($NaNO_2$)	Blank in P&O Gallery
	FG126 Steam to Jet F10 to F13	Lock & tag in Control Rm.
Tank F13	FG66 Steam to Jet F13 to F16-G1	Lock & tag in Control Rm.
	FG70 Power to Pump F13 to F14	Lock-out pump in Motor Control Station
Tank F16	FG37 Steam to Jet F16 to E3	Blank in P&O Gallery
	FG36 Steam to Jet F16 to F13	Blank in P&O Gallery
	FG31 Steam to Jet F16 to F8	Lock & tag in Control Rm.

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Vessel	Nozzle or Line	Action
Tank F18	FG10 Steam to Jet F18 to F13	Lock & tag in H.E. Control Rm
Tank G1	GG164 Steam to Jet G1 to G5, G7	Lock & tag in Control Rm.
	GT111 Jet G1 to F13	Steam jumper currently removed
Column T-G2	GG129 Steam to Jet TG2 to G2 (MT out)	Lock & tag in Control Rm.
Tank G2	GG116 Steam to Jet G2 to UGS	Lock & tag in Control Rm.
	GG127 Na_2CO_3 Add to G2	Blank in P&O Gallery
Tank G5	GG67 Steam to Jet G5 to G1	Lock & tag in Control Rm.
Tank G8	GG7 Steam to Jet G8 to F16	Lock & tag in Control Rm.
	GT1 G8 to F7	Lock & tag in Control Rm.
Tank H1	HG91 Chem Add	Blank in P&O Gallery
	HG93 Steam to Jet H1 to E6	Lock & tag in Control Rm.
Column T-H2	HG52 Steam to Jet H2 to F7 (MT out)	Lock & tag in Control Rm.
	HT40 3WB to H2	Blank nozzle in cell
	Column Nozzles V, C, R	Blank nozzles in column not in use
	HG59 Chem Add	Blank 7007 Reader to nozzle in P&O Gallery
Column T-H3	HG42 Steam to Jet H3 to F13	Lock & tag in Control Rm.
Tank J21	JG138 Steam to Jet J21 to F10	Lock & tag in Control Rm.
Column T-J22	JG157 Steam to Jet J22 to F10	Lock & tag in Control Rm.
Column T-J23	JG159 Steam to Jet J23 to F10	Lock & tag in Control Rm.
Tank J3	JG131 Steam to Jet J3 to F13	Lock & tag in Control Rm.
Column T-J4	JG98 Air to 1BP DOV	Lock & tag in Control Rm.
	JG100 Steam to Jet J4 to J5	Lock & tag in Control Rm.
Column T-J6	JG66 Steam to Jet J6 to F13 (MT out)	Lock & tag in Control Rm.
Column T-J7	JG37 Normal 1CX to J7	Blank in P&O Gallery
	JG42 Steam to Jet J5 to F13	Lock & tag in Control Rm.
Tank K1	KG120 HNO_3 to K1	Blank in Sample Gallery
	KG127 Steam to Jet K1 to K6	Lock & tag in Control Rm.
	KG129 Steam to Jet K1 to J8	Lock & tag in Control Rm.

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Vessel	Nozzle or Line	Action
Column T-K2	KG88 Steam to Jet 2D Col. to F10	Lock & tag in Control Rm.
	KG95 HNO_3 to 2DS	Blank in P&O Gallery
Column T-K3	KG75 Steam to Jet K3 to F13	Lock & tag in Control Rm.
Tank K6	KG16 Chem Add	Blank in White Room
	KG19 Steam to Jet K6 to F13	Lock & tag in Control Rm.
	KG10 Steam to Jet K6 to E6	Tag (transfer to be controlled by supervisor)
	KG11 Steam to Jet K6 to N1	Tag (transfer to be controlled by supervisor)
Column T-L1	LG68 Steam to Jet L1 to F10 (MT out)	Lock & tag in Control Rm.
	LG78 Alt. #2 Org. 2AX	Blank in Sample Gallery
Column T-L2	LG51 Steam to Jet 12 L2 to F10 (MT out)	Lock & tag in Control Rm.
	3BU Valve (2BP normally)	Tag to route to N1 only
Tank R1	Steam to Jet R1 to G1	Lock & tag in Control Rm.
	Steam to Jet R1 to F13	Lock & tag in Control Rm.
Tank R2	Steam to Jet R2 to G2	Lock & tag in Control Rm.
Tank R8	Steam to Jet R8 to F16	Lock & tag in Control Rm.
	Steam to Jet R8 to F7	Lock & tag in Control Rm.
Tanks U1 & U2	Blank header at following location in Sample Gallery: U5 A3 B3 C3	
Tank N1	Steam to TK-N1 Preheater	Blank at preheater
XAW Sample Pot	Line from XAW Sample Pot to XAW Jet	Blank to include XAW and XSW
Resin Reservoir	Line from Resin Reservoir to XAW Jet	Blank
Tank L9(L10)	PR Can Slurp Line to L9 (L10) Sample Tank.	Blank

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3.10 Chemical Hazards

3.10.1 General

Because of the fact that the chemicals (essential materials) used for thorium and normal uranium processing are very nearly the same, the bases for control of chemical hazards during thorium processing are the same as those presented for normal uranium processing(1). The chemicals to be used for thorium processing are listed in Section 4.3.1 of this document. Those that are new, as compared to normal uranium processing requirements, are potassium fluoride and phosphoric acid. With respect to potential hazards, these are similar to sodium fluoride and nitric acid, respectively, and should be handled with the same precautions.

In any process using noncompatible chemicals in different portions of the flowsheet, care must always be taken to isolate the chemicals in the chemical storage and make-up areas. Transfer lines to the process areas must be isolated in order to prevent line mixing. Listed below are a few general precautions that should be taken during the thorium run.

1) Isolate chemical make-up systems. (This is covered under Section 3.9 concerned with line blanking.)
2) Make certain the correct chemicals are used in chemical make-up.
3) Store hydrazine in cool place and do not mix with hot concentrated nitric acid solutions.
4) Make certain chemical headers are flushed and isolated prior to use.

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(1) R. G. Geier, "Process Specifications for Chemical Hazards Control - Purex Plant," HW-67757, December 1960 (Unclassified).

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For further information, References (1) and (2) and others give specific information about the chemicals to be used in the thorium run.

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(1) R. G. Geier, "Process Specifications for Chemical Hazards Control - Purex Plant," HW-67757, December 1960 (Unclassified).

(2) Reinhold Publishing Corporation, New York, "Dangerous Properties of Industrial Materials," 1957, N. I. Sax.

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3.10.2 Heat Generation in Thorium Solutions

Heat generation in thorium solutions to be processed in Purex depends upon reactor history and cooling time. For the material to be processed during the thorium run most (>85%) of the heat generated will come from protactinium-233 (half life 27.4 days). Since there will be no protactinium recovery during the run, the highest concentration of protactinium will occur in the waste concentration system (1WW) under normal flowsheet conditions.

For normal flowsheet conditions, all Purex vessels have adequate cooling capacity to maintain the solution temperature below 50 C; however, as an aid in calculating heat generation for abnormal situations, the following information is given:

1) The maximum heat generation rate on June 1, 1966 will be 14,200 BTU/hr/ton thorium (based on a 17-ton thorium discharge on February 25, 1966 from the KE core). Assuming the waste from processing material of this type were collected in a 1WW storage tank (Tanks F7, F15, or F16), the maximum heat generation rate would be 380,000 BTU/hr, or approximately 55 per cent of the heat generated from a tank full of waste produced from 120-day cooled normal uranium. More than half of the thorium to be processed will have been cooled longer than 225 days on June 1, 1966 and will therefore have very little heat content.

2) Listed below are the cooling capabilities of pertinent Purex vessels:

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Vessel	Cooling Capabilities*
a) Standard Purex Vessel (TK-D1, D2, D5, E3, E5, E6 F3, F4, F7, F5, F10, F12, F13, F15, F16, F18, G8, H1, J1, J3, K1, K5, K6)	>500,000 BTU/hr
b) Tanks E1, G2	>150,000 BTU/hr
c) C3 Pot Dissolver	>900,000 BTU/hr
d) Purex Annular Dissolver	>1,000,000 BTU/hr

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*Assumes 40° solution temperature and a cooling water temperature of 20° C and a heat transfer coefficient of 50 BTU/hr-ft^2-° F.

[redacted]

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3.10.3 Radiolytic Hydrogen Formation in Thorium Solutions

Hydrogen is formed in radioactive solutions at a rate of 0.043 cubic feet per kilowatt hour (12 cubic centimeter per watt hour) (1). Under flowsheet conditions during the thorium run, the maximum rate of formation of hydrogen will occur in the waste storage tanks (TK's F7, F15, F16) at a rate of 4.8 cubic feet per hour*. Air dilution of approximately 240 cubic feet per hour will be required to maintain a safe hydrogen concentration of less than 2 volume per cent. Air dilution has been shown to be adequate for normal uranium processing when there is ~ 2 times as much radiolytic heat in the tanks.

It is recognized that in case of failure of the vessel vent vacuum, hydrogen could build up in tanks containing large amounts of radiolytic energy. For tanks containing greater than 3500 watts of radiolytic energy, Reference (2) recommends de-energizing, locking, and tagging all electrical equipment associated with the tank if the vessel vent system fails. The equipment should remain off until the vent vacuum has been on at least 20 minutes. This procedure should be followed if possible during the thorium run. If the vessel vent system fails, the instrument air to the tank should be turned to maximum rate to provide as much air dilution as possible.

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*Based upon 17 tons thoria discharged from KE on Feb. 25, 1966, which contained the largest amount of Pa-233 per ton of any thorium to be processed.

(1) L. P. Bupp, "Chemical Research and Development Report," HW-69063, March 12, 1961, (Secret)

(2) Letter, W. C. Schmidt and B. F. Judson to R. W. McCullugh and S. G. Smolen, "Radiolytic Hydrogen in Purex and Strontium Semiworks," dated December 12, 1962.

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SECTION 4

PROCESS SPECIFICATIONS

4.0 Introduction

4.1 Feed Specifications

4.2 Product Purity Goals*

4.3 Essential Materials Specifications

4.4 Critical Mass Control Specifications

4.5 Chemical Hazards Control Specifications

*) At the present time, customer requirements for product purity have not been defined; hence actual product specifications cannot be written at this time.

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4. PROCESS SPECIFICATIONS

4.0 INTRODUCTION

The process specifications for thorium processing operations in the Purex Plant are presented in this section. On the basis of the thorium processing flowsheet, Section 2 of this document, these specifications represent currently known limits within which plant processing conditions must be maintained to meet defined product requirements safely and with minimum effect on equipment service life. The process specifications cover the general areas of (1) feed, (2) essential materials, (3) critical mass, and (4) chemical hazards. While specifications should also ordinarily be provided for uranium-233 and thorium products, at the present time the customer specifications do not exist; consequently the product quality criteria presented under 4.2 of this section should be considered as "best effort" or "target" values that should be achieved, rather than as specifications.

The approvals required for these specifications are designated by the individual approval sheets at the end of each specification sub-section. For any specification changes that may be required, it is intended that the designated level of approval also be required. For each specification sub-section, a separate sheet will be provided for recording and approving such changes.

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4.1 Feed Specifications

4.1.1 On the basis of reactor shipping records and any other reasonable control method, no reactor product materials other than DUN reactor, aluminum-clad, irradiated thoria targets shall be accepted for processing at the Purex Plant during a scheduled thoria processing campaign.

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4.1.2 Scheduling of the charging of the irradiated thoria target elements to the dissolvers, as based on reactor shipping records, shall conform to the following specifications:

a. Charging of the elements to the dissolver shall be scheduled to continuously produce a Purex U-233 product blend with a U-232-to-U-233 mass ratio of less than 6×10^{-6}.

b. The combined concentrations of uranium-233 and protactinium-233 per dissolver charge shall not average greater than 2200 grams per ton of thorium.

c. The concentration of I-131 per dissolver charge shall not average greater than 20 curies per ton of thorium*.

d. The concentration of Pa-233 per dissolver charge shall not average greater than 50 grams per ton of thorium.

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* The intent of this specification is to insure that the emission of I-131 from the Purex Plant can be practicably controlled at less than 0.2 curies per day.

[redacted]

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4.1.3 Authorizations

Prepared By: Jack B Kendall Date: 3-1-66
Process Engineer, Separations Process Engineering

Issued By: Hal C. Rathvon Date: 3-1-66
Manager, Separations Process Engineering

Accepted By: Lyle L. Zahn, Jr. Date: 3-3-66
Manager, Purex Processing

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4.2 Product Purity Goals

The capability of the thorium processing flowsheet for consistently producing uranium-233 and thorium nitrate products of specified quality has not been demonstrated. Hence, firm product specifications have not been defined. Instead, purity goals are proposed on the bases of (a) process performance estimates by Separations Process Engineering, and (b) estimates of requirements by potential customers. These proposed goals should also provide product of adequate purity for any on-site processing that might be subsequently planned.

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4.2.1 General

a. These goals require no formal approval. Additions and revisions shall be made by mutual agreement of the Manager, Purex Processing and the Manager, Separations Process Engineering.

b. Failure to meet any of these goals shall be investigated and the cause corrected, as possible, to the mutual satisfaction of the Manager, Purex Processing, and the Manager, Separations Process Engineering.

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4.2.2 Uranium-233 Product

The proposed sampling and analytical requirements and purity goals for the uranium-233 product are presented in this section.

a. Sampling

A 60 to 80 ml sample of each batch of product solution should be obtained from the U-233 Load-Out Tank and transported to the analytical laboratory immediately following transfer of a batch of product solution from the Product Receiver (TK-N7) to the U-233 Load-Out Head Tank. An aliquote sufficient for the Purex analytical requirements should be removed and the remainder sent directly to the 234-5 laboratory (or retained for compositing with samples from other batches, as appropriate) for mass spectrometric and emission spectrographic analyses.

b. Analytical

The suggested analytical methods are presented in the table below. Other more suitable methods might be developed and may be used. However, the Manager, Separations Process Engineering, should be advised of any such changes.

c. Product Quality

Component	Target Limit*	Suggested Analytical Method
Uranium-233	300 to 375 g/l	X-ray
HNO_3	1.0 ± 0.5 lb/gal	HpH
Uranium Isotopes		
U^{232}	8 ppm, (U^{233} basis)	Alpha Energy
U^{233}	95%, Minimum	Mass Spectrometry
U^{234}	2.5%	"
U^{235}	0.5%	"
U^{236}	0.1%	"
U^{238}	3.9%	"

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*Maximum, unless otherwise indicated.

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c. Product Quality (Cont'd)

Component	Target Limit*	Suggested Analytical Method
Other Radiochemical Impurities (U^{233} basis)		
Pu	0.001 g/g	Pu A-30a
Th	0.001 g/g	Th Sep-1a
Np	0.0003 g/g	Po A-6a
ZrNb-95	2 μ Ci/g	E-2a
Ru-103, RuRh-106	2 μ Ci/g	E-2a
Pa	8 μ Ci/g	U-1 x , γ scan
Chemical Impurities		
Aluminum	200 ppm	Emission Spectrography
Arsenic	40 ppm	"
Boron	1 ppm	"
Cadmium	2.5 ppm	"
Calcium	80 ppm	"
Chromium	350 ppm	"
Copper	40 ppm	"
Iron	1500 ppm	Fe S-1e
Lead	300 ppm	Emission Spectrography
Magnesium	100 ppm	"
Manganese	40 ppm	"
Molybdenum	450 ppm	"
Nickel	200 ppm	"
Phosphorous	100 ppm	"
Potassium	40 ppm	"
Silicon	250 ppm	"
Sodium	60 ppm	Flame Photometer
Vanadium	80 ppm	Emission Spectrography
Zinc	80 ppm	"

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*Maximum, unless otherwise indicated.

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4.2.3 Thorium Product

The proposed sampling and analytical requirements and purity goals presented in this section should provide a TNT product of adequate purity for recycling to the reactors.

a. Sampling

A bottle sample (150-200 ml) should be obtained from each batch of TNT product in TK-K6. An aliquot sufficient for the Purex analytical requirements is removed and the remainder held for compositing. The number of batches to be composited will be determined.

b. Analytical

The suggested analytical methods presented below reflect the most satisfactory methods at publication of this document. Any improved methods subsequently developed may be used, but the Manager, Separations Process Engineering should be so advised.

c. Product Quality

Component	Target Limit	Suggested Analytical Method
Thorium	3.5 ± 0.5 lb/gal	Th T-1a
HNO_3	0.5 ± 0.3 lb/gal	HV-4a or HpH
Radiochemical Impurities (Thorium weight basis):		
Pu	≤ 10 ppb	Pu A-20a
U-233	≤ 20 ppm	U-1x
Total U(excluding U-233)	≤ 10 ppm	UF
ZrNb-95	≤ 50 u Ci/lb	E-2a
Ru-103, RuRh-106	≤ 50 u Ci/lb	E-2a
Pa-233	≤ 300 μ Ci/lb	E-2a
Chemical Impurities (Thorium weight basis):		
Silicon	≤ 50 ppm	Emission Spectrography
Iron	≤ 100 ppm	Emission Spectrography
Total Impurities*	≤ 1000 ppm	Emission Spectrography
Chloride	≤ 50 ppm	To be determined
Sulfate	≤ 200 ppm	To be determined

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* Al, B, Be, Bi, C, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, Pb, Si, Sn, Zn

[redacted]

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4.3 Essential Materials Specifications

4.3.1 The essential materials required for thorium processing are listed below. Except for potassium fluoride (KF) and phosphoric acid (H_3PO_4), the specifications for these materials shall be as defined by the Essential Materials Manual.(1)

a) Sodium hydroxide (50% solution)
b) Nitric Acid (57% solution)
c) Aluminum nitrate nonahydrate
d) Ferrous sulfamate
e) Phosphoric Acid
f) Sugar (sucrose)
g) Normal Paraffin Hydrocarbon
h) Tributyl phosphate
i) Hydrazine
j) Sodium fluoride
k) Sodium Nitrite
l) Sodium Carbonate
m) Potassium permanganate
n) Potassium fluoride

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(1) HW-54125 (Unclassified), "Essential Materials Manual," Research and Engineering, Chemical Processing Department, October 1, 1958.

[redacted]

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4.3.2 Essential materials, other than those listed under 4.3.1, above, shall not be introduced to any thorium processing stream without the approval of the Manager, Separations Process Engineering.

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4.3.3 The potassium fluoride is specified as technical grade and the phosphoric acid as food grade, NF grade or reagent grade. The following acceptance specifications shall also be met.

a. Potassium Fluoride (Anhydrous Powder)

Material	Value	Analytical Method	Sample Frequency
KF	Min. 95 Wt. %	F-2	Each shipment
Chloride	Max. 0.1 Wt. %	CL-5	Each Shipment

b. Phosphoric Acid (Aqueous Solution)

Material	Value	Analytical Method	Sample Frequency
H_3PO_4	Min. 85 Wt. %	H-1	Each Shipment
Chloride	Max. 0.1 Wt. %	CL-5	Each Shipment

[redacted]

[redacted]

4.3.4 Authorizations

Prepared By: S M Nielson Date: 9/1/65
Process Engineer, Separations Process Engineering

Issued By: H. C. Rathvon Date: 9/1/65
Manager, Separations Process Engineering

Accepted By: Date: 3-3-66
Manager, Purex Processing

[redacted]

[redacted]

4.4 CRITICAL MASS CONTROL SPECIFICATIONS

The specifications below define the limits or restrictions necessary to maintain the probability of a criticality incident in the Purex Plant at acceptably low levels while processing irradiated thoria. They were developed in conjunction with the other specifications included in this document and in conjunction with the chemical processes that have been defined and recommended (Sections 2.0 and 3.0) for processing the specified feed material (Section 4.1).

These critical mass control specifications do not stipulate the system, procedures, or mechanisms to permit operation within the limits or restrictions. An administrative system composed of written procedures, calculated material balances, stipulated sampling sequence and frequency, and other measurement and control techniques may be used to augment available in-line monitors and direct reading instruments.

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4.4.1 General

a. Additions and revisions to these specifications require the same levels of approval as the specifications themselves.

b. The process vessels covered by these specifications are named within the specifications. Process vessels include both process equipment pieces and process sumps.

c. Process vessels which may contain fissionable materials and for which controls are not defined by these specifications shall be governed by the specifications in HW-71625(1), or by any specifications which shall supersede HW-71625, in whole or in part. For purposes of applying these specifications, any U-233 or Pa-233 in these vessels shall be considered to be Pu-239.

d. Physical changes to the Purex Plant processing system required for thorium - uranium-233 processing, as defined by detailed design drawings, shall be approved by the Manager, Separations Process Engineering, or his designated representative, prior to the introduction of uranium-233 to the processing system. Following such approval, no changes shall be made to any of the following systems that would invalidate as-built drawings or other accepted descriptive material without the prior approval of the Manager, Separations Process Engineering:

J-Cell Package
L Cell
N Cell
PR Room
1C Column (T-H3)
1CU Concentrator (E-H4)
2AF Tank (TK-J21)
3AF Tank (TK-J5)
U-233 Product Containers and Carriers

e. The plutonium-239 content of any vessel covered by these specifications, and containing uranium-233, protactinium-233, and/or thorium in any combination shall be restricted to a maximum of 250 grams. For purposes of applying any other limit imposed by the specifications of this document, any plutonium-239 content in any of the process vessels shall be considered to be uranium-233.

f. Additions of uranium-233 or protactinium-233 to any process vessels not covered by these specifications shall require the approval of the Manager, Separations Process Engineering.

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(1) B. F. Judson and G. C. Oberg, "Process Specifications for Critical Mass Control - Purex Plant (Revision No. 3)," HW-71625, October 15, 1962 (Secret).

[redacted]

g. Precipitating agents, such as caustic and oxalic acid, shall not be combined in any way with a mass greater than 500 grams total, of U-233, Pa-233, and Pu-239, unless sufficient thorium is present to prevent exceeding a ratio of 2200 grams, total, of U-233, Pa-233, and Pu-239 per ton of thorium. Violation of this paragraph shall be considered to be a case, but not the only possible case, of uncontrolled precipitation.

h. Any indications of uncontrolled accumulation, precipitation, or mixing of normal uranium, thorium, protactinium-233, uranium-233, or plutonium in the process system shall be referred immediately to the Manager, Separations Process Engineering. In the event that uncontrolled accumulation, precipitation, or mixing is confirmed, the difficulty shall be corrected in accordance with procedures approved by the Manager, Separations Process Engineering, and in accordance with supplemental specifications as required.

i. Any indications of the uncontrolled accumulation of Pa-233, U-233, and/or Pu-239 in a solvent phase shall be referred immediately to the Manager, Separations Process Engineering.

j. The minimum and maximum tributyl phosphate concentrations of the HAX, 1BS, 2AX, and 3AX streams shall be 28 and 32 volume per cent, respectively.

k. A minimum routine sample schedule, issued by the Supervisor, Purex Process Control, shall be approved by the Manager, Separations Process Engineering, for criticality control considerations. Any revisions to the routine sample schedule which would reduce the specified minimum sampling frequency are also subject to approval for criticality control considerations. It is the intent of the specifications of this paragraph that a schedule of sampling and analysis be followed that will insure adequate criticality control, as judged by the Manager, Separations Process Engineering.

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4.4.2 Feed Preparation

The specifications in this section apply to the following vessels: A3 (Dissolver), B3 (Dissolver), C3 (Acid Boil-Off), TK-D1 (Acid Boil-Off Feed), TK-D2 (Coating Waste), TK-D5 (Stock TNT), TK-E1 (Centrifuge Product), G-E2 (Coating Waste Centrifuge), TK-E3 (Centrifuge Feed), G-E4 (Coating Waste Centrifuge), TK-E5 (Centrifuge Waste), TK-E6 (HAF Make-up), and TK-H1 (HAF).

a. No more than 10 tons of irradiated thoria shall be present in a Purex thoria dissolver (A3 and B3).

b. The maximum combined concentration of uranium-233 and protactinium-233 shall be 2200 grams per ton of thorium.

c. For purposes of controlling transfers of U-233 from TK-L11, the following additional limits shall apply to TK-E6 during such transfers:

   (1) Tank E6 shall contain at least 2000 gallons of solution with a minimum thorium concentration of 0.5 M.

   (2) Both agitators in TK-E6 shall be operating.

   (3) The maximum combined concentrations of uranium-233, protactinium-233, and plutonium-239 in a solution added to TK-E6 shall be 227 g/gal (60 g/l)*.

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* The L11-to-E6 transfer jet dilutes the L11 concentration four fold. Therefore, to meet this specification, the concentration in TK-L11 must be $\leq$910 g/gal (240 g/l) prior to the transfer.

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4.4.3 Co-Decontamination and Partition Cycle

a. The concentration and mass of U-233 in TK-J3 (1BXF Tank) shall be limited as follows:

(1) The maximum concentration of U-233 in TK-J3 shall be 3000 grams per ton of thorium. (Note: This limit will insure operation within the desired limit in the HA Column, T-H2).

(2) The maximum concentration of U-233 in TK-J3 shall be less than 0.45 g/gal (0.12 g/l).

b. The concentration and mass of U-233 in TK-J2 (HAO Tank) shall be limited as follows:

(1) The maximum concentration of U-233 in TK-J2 shall be 9.5 g/gal (2.5 g/l). (Note: This limit will insure operation within the desired concentration limit of 9.5 g/gal (2.5 g/l) in the 1BS Column, T-J7).

(2) The maximum mass of U-233 in TK-J2 shall be 3 Kg.

c. The maximum U-233 concentration in the 1BX Column T-J6 shall be limited to 9.5 g/gal (2.5 g/l) as follows:

(1) The HNO_3 concentration in the 1BX stream shall not be less than 0.12 M. This limit shall also apply to any aqueous phase introduced to T-J6 in connection with the startup of processing.

(2) The T-J6 aqueous-to-organic flow ratio shall not exceed 0.7.

(3) The maximum temperatures of the 1BX and 1BXF streams shall be 35° C.

(Note: The 1BX acidity should not exceed 0.2 M HNO_3, the minimum aqueous-to-organic flow ratio in the 1BX Column should be 0.5, and the minimum 1BX Column temperature should be 25° C in order to adequately strip thorium from the 1BU stream.)

d. The 1C Column (T-H3) aqueous-to-organic flow ratio shall be greater than 0.45. (Note: This limit will insure operation within the desired concentration limit of 20.8 g/gal (5.5 g/l) in T-H3.)

e. All unused process lines except steam, cooling water, electrical, and air lines from the P & O Gallery and the Sample Gallery to the HA Column (T-H2), 1C Column (T-H3), HAO Tank (TK-J2), 1BXF Tank (TK-J3), T-J4, 1BX Column (T-J6), and 1BS Column (T-J7) shall be blanked during uranium-233 processing.

[redacted]

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4.4.4 Second Uranium Cycle

a. The maximum masses of uranium-233 in the 1CU Concentrator (E-H4), 1UC Receiver (TK-J1), and 2AF Tank (TK-J21), shall be 9.0, 9.0, and 3.5 kilograms, respectively. In addition, the concentration of uranium and nitrate in the aqueous phases of E-H4, TK-J1, and TK-J21 shall be controlled within the limits (Permitted Region) shown in Figure 4.4-I.

b. The concentration of U-233 in the 2A Column (T-J22) and in the 2B Column (T-J23) shall be maintained at less than 30 g/gal (8 g/l) as follows:

(1) The HNO_3 concentration in the 2AS stream shall be not less than 0.1 M. This limit shall also apply to any aqueous phase introduced to T-J22 in connection with the startup of processing.

(2) The maximum U-233 concentration in the 2AF Tank (TK-J21) shall be 3.6 g/gal (0.95 g/l).

(3) The maximum 2AF-to-2AX flow ratio shall be 5.0.

(4) The minimum 2B Column aqueous-to-organic flow ratio shall be 0.6.

c. All unused process lines from the Sample Gallery and the P&O Gallery to the 1CU Concentrator (E-H4), 1UC Receiver (TK-J1), 2AF Tank (TK-J21), 2A Column (T-J22), and 2B Column (T-J23) shall be blanked during uranium-233 processing except steam, cooling water, electrical, air, and the seal pot water to E-H4.

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4.4.5 Third Uranium Cycle

a. The maximum mass of uranium-233 in the 3AF Tank (TK-J5) shall be 1500 grams. In addition, the concentrations of uranium and nitrate in the aqueous phases of 3AF Tank (TK-J5) and 3BU Receiver (TK-N1) shall be controlled within the limits shown in Figures 4.4-II and 4.4-III (Permitted Region), respectively.

b. The concentration of U-233 in the 3A Column (T-L1) and in the 3B Column (T-L2) shall be maintained less than 152 g/gal (40 g/l) as follows:

  (1) The HNO_3 concentration in the 3AS stream shall be not less than 0.08 M. This limit shall also apply to any aqueous phase introduced to T-L1 in connection with the startup of processing.

  (2) The maximum U-233 concentration in the 3AF Tank (TK-J5) shall be 20.8 g/gal (5.5 g/l).

  (3) The maximum 3AF-to-3AX flow ratio shall be 5.0.

  (4) The minimum 3B Column aqueous-to-organic flow ratio shall be 0.7.

c. The maximum concentration of uranium-233 in the Fission Product Adsorption Column and the Thorium Adsorption Column shall be 755 g/gal (200 g/l). Note: The maximum possible uranium loading on either column is 750 g/gal (198 g/l) of resin.

d. All unused process lines from the P&O Gallery and the Sample Gallery to 3AF Tank (TK-J5), 3A Column (T-L1), 3B Column (T-L2), and 3BU Receiver (TK-N1) shall be blanked during uranium-233 processing except the steam, cooling water, electrical and air lines.

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4.4.6 Second Thorium Cycle

a. The maximum concentration of uranium-233 in the Second Thorium Cycle vessesl [1BT Concentrator (E-J8), 2DF Tank (TK-K1), 2ET Concentrator (E-K4), 2TC Receiver (TK-K5), and TNT Product Tank (TK-K6)] shall be 30 grams per ton of thorium.*

b. The maximum combined masses of uranium-233 in the 2D Column (T-K2) and 2E Column (T-K3) shall be 500 grams.

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* This limit insures that the mass limit for TK-R1 (500 grams; see 4.4.7, below) will not be exceeded in the event of a loss of uranium via the 2EW stream. Except for this consideration, the allowable U-233 in the Second Thorium Cycle vessels would be at least 2200 grams per ton of thorium.

[redacted]

4.4.7 Solvent Recovery Cycles

a. The maximum mass of uranium-233 in any one of the G and R Cell vessels shall be 500 grams.

[redacted]

4.4.8 Waste Concentration

a. The maximum combined masses of uranium-233, protactinium-233 and plutonium-239 in any one of the waste treatment vessels (E-F6, TK-F7, TK-F10, TK-F12, TK-F13, TK-F15, TK-F16, and TK-F18) shall be 7.0 kilograms. For cases in which caustic will be added to any one of these vessels, the maximum combined masses of uranium-233, protactinium-233, and plutonium-239 in that vessel shall be 500 grams.

[redacted]

4.4.9 Acid Recovery

a. The maximum mass of uranium-233 and plutonium-239 in any one of the acid recovery vessels (TK-F3, T-F5, U-Cell vessels, and Back-up Acid Recovery vessels) shall be 500 grams.

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4.4.10 Sumps

a. The maximum combined concentrations of uranium-233 and plutonium-239 in any sump shall be 20.8 g/gal (5.5 g/l) unless specified otherwise.

b. The maximum combined concentrations of uranium-233 and plutonium-239 in the SLA Sump shall be 1700 g/gal (450 g/l). The maximum depth of solution from the lowest point of the SLA Sump shall be 9 inches.

c. The maximum combined concentrations of uranium-233 and plutonium-239 in the SLE, SLF, and SLD Sumps shall be 450 grams per liter.

The maximum depth of solution from the lowest point of the SLE and SLF Sumps* shall be one inch.

The maximum depth of solution from the lowest point of the SLD Sump shall be three inches**.

d. The maximum combined concentrations of uranium-233 and plutonium-239 in the SN Sump* shall be 1700 g/gal (450 g/l). The maximum depth of solution from the lowest point of the SN Sump shall be 1-3/4 inches.

e. The maximum combined concentrations of uranium-233 and plutonium-239 in the SE Sump shall be 227 g/gal (60 g/l).

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*The specifications indicated do not require that the SLF, SLE, SLD, and SN Sump be free of debris or that the drain lines from SLE and SLF to SLD, and from SLD to SLA be unrestricted. However, in the interests of insuring control at the solution depths specified, it is recommended that sumps be maintained clean, with unrestricted flow through drains that are provided.

**This specification insures sufficient remaining volume in SLD Sump to safely contain the contents of any tank that might fail and drain to that sump. The volume calculation assumes the addition of poison Raschig Rings of 65 per cent void volume to the existing sump with its Boral grid. A poison system of different volume would require (or permit) adjustment to this value. For example, an increase in Raschig Ring void volume would increase the volume available to contain a tank failure; an increase in the maximum solution depth could thereby be justified.

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4.4.11 L-Cell Package, PR Room, and N-Cell

a. The maximum combined concentrations of uranium-233 and plutonium-239 in T-L3, E-L4, TK-L6, TK-L9, TK-L10, and TK-L13 shall be 280 g/gal (75 g/l).

b. T-L3 and E-L4 vessels shall not be used for concentrating uranium-233 solutions.

c. Entry of personnel into the L-Cell or into the N-Cell Hood, SLD Hood, PR Room Hoods, and U-233 Load-out Hood, except for hands and arms, shall be made in accordance with procedures approved by the Manager, Separations Process Engineering, and in accordance with supplemental specifications, as required.

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4.4.12 Product Handling - Containers and Carriers

a. Definitions

(1) As used in these specifications, product containers and carriers approved for storage and transport of uranium-233 solutions are defined as follows:

Type A*

Container: Polyethylene bottle; capacity: one gallon, Stock #57-5155-035.

Carrier (Type A-1): M-102 "Bird Cage" (Dow Chemical Co., Drawing #12720-14 Rev.) which has been modified to include a stainless steel insert and retainer ring for secondary containment.

Type B*

Container: Polyethylene bottle, nominal 3 liter capacity (actual 3.3 to 3.7 liters); defined by Drawing H-2-32973.

Carrier: Storage (Type B-1), 30-gallon steel drum provided with a stainless steel insert for secondary containment. The annular space (about 7 inches thick) is concrete filled. Details are defined by Drawing H-2-32920.

Carrier: Shipping (Type B-2), Westinghouse; 55-gallon steel drum with a stainless steel insert for secondary containment. The annular space is filled with Vermiculite. Details are defined by Drawing #966 D873, Westinghouse Electric Corporation, Bettis Atomic Power Laboratory.

Carrier: Shipping (Type B-3), ORNL; 55-gallon steel drum with a 5-inch, schedule 80 stainless steel pipe insert for secondary containment. The void space is filled with foamed glass. Details are defined by Drawing #D-58137, Union Carbide Nuclear Company

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*The letter and number designations (e.g., Type A, B-2, etc.) were adopted for convenience in defining the containers and carriers described herein. They have no other significance.

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(2) As used in these specifications, an array is a single tier horizontal arrangement of any number of Type A or B containers within Type A-1, B-1, B-2 or B-3 carriers respectively (including any in transit) in which the spacing between any two adjacent carriers is less than 12 feet, edge-to-edge, and in which the neutron shielding equivalent of any material separating any two adjacent carriers is less than 12 inches of concrete.*

b. Container Limits

Container	Max. Conc'n. g U-233/l	Max. Mass g U-233/container
Type A	75	280
Type B	450**	1400**

(1) Only one loaded Type A or Type B container shall be outside its carrier at a time.

(2) At any time during which operations require the removal of a Type A or B container from its carrier, the distance between a bare container and an adjacent carrier shall be limited by design or operation of handling facilities to a minimum of ten inches, edge-to-edge. These containers shall not be passed over any carrier.

(3) If uranium-233 container-carriers of different types are stored in a mixed common array, the array limits for the most restrictive case shall apply to the entire array.

(4) The spacing between a uranium-233 container and a container of any other type fissionable material shall be at least 12 feet distance in air; alternatively, the containers may be separated by at least 12 inches of concrete or its neutron-shielding equivalent of other materials.

(5) No limit is imposed on the number of container-carriers stored in a single or double-line array.

(6) Arrays of Type A container-carriers, other than single or double-line arrays, shall be limited to not more than 30 container-carriers.

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*Under the scope of this definition, the transport of Type A or B container(s) within Type A-1, B-1, B-2 or B-3 carrier(s) between two arrays might require that the total number of carriers (in the two arrays plus the one(s) in transit) be considered as a single large array.

**Shipping requirements are 375 g/l maximum and 3 liters maximum.

[redacted]

[redacted]

(7) Type B containers may be stored in Type B-1 carriers without limit as to the number of containers in the array.

(8) Arrays of Type B containers in Type B-2 or B-3 carriers, other than single or double line arrays, shall be limited to 36 containers.*

(9) The product containers, including secondary containers, shall be vented during storage.**

(10) The secondary containment vessel of each shipping or storage carrier shall be sealed while in transit when the carrier contains uranium-233 solutions.

c. Identification of Product Containers

(1) Immediately following the addition of material to a product container, the material shall be identified by attaching a tag or label to the container and to the carrier.

d. Lids on Carriers

(1) Lids shall be securely fastened on all carriers, except that one loaded carrier and one empty carrier may be opened, at one time, for loading, unloading, transfer or inspection.

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*This specification pertains only to on-site product handling under CPD supervision.

**Either continuous or intermittent venting is acceptable. Intermittent venting should be scheduled at a minimum frequency of one week. The special vent cap provided for the Type B containers is considered to continuously vent the container.

[redacted]

4.4.13 Storage and Transportation of Samples and Laboratory Solutions

a. Definitions

(1) Process Samples

For purposes of this section, a process sample may be a single sample or a composite of liquid and/or solid materials.

(2) Disposal Bottle

For purposes of this section, a disposal bottle shall have a maximum diameter of 4.8 inches and a maximum volume of 3.7 liters. The bottle defined by Drawing H-2-32973 meets these requirements. Other bottles that meet these requirements may be used.

(3) Work Station

For purposes of this section, a work station is any hood, group of hoods, cabinet or other identifiable zone that contains more than 500 grams of uranium-233.

(4) Laboratory Solution

For purposes of this section, laboratory solutions are considered to consist of the solution in a sample disposal bottle with a uranium-233 concentration in excess of 6 grams per liter.

(5) For purposes of this section, the Purex Plant is considered to consist of two locations: the Purex Analytical Laboratory and the rest of the plant.

(6) Sample Storage Station

For purposes of this section, the Sample Storage Station is a single cabinet in the Purex Analytical Laboratory used for storing sample and laboratory process materials.

b. Limits: Process Samples

(1) Any single process sample shall be limited to a maximum volume of 500 milliliters unless the uranium-233 concentration in the sample is less than 23 grams per gallon (6 grams per liter) as determined by analysis. In this case, the maximum volume shall be 41 liters.

(2) No single process sample shall contain greater than 250 grams of uranium-233.

c. Limits: Disposal Bottles

The maximum concentration of uranium-233 in a disposal bottle shall be 450 grams per liter.

The maximum mass of uranium-233 in a disposal bottle shall be 1400 grams.

d. Limits: Purex Analytical Laboratory*

(1) Work Station

The maximum number of disposal bottles within a work station shall be one.

The maximum total volume of all containers other than the disposal bottle in the work station shall be 1.3 liters.

The reflection around any disposal bottle or container shall not exceed the equivalent of one inch of water.

Work stations shall be separated from each other and from the Sample Storage Station by 12 feet, edge-to-edge, or the neutron shielding equivalent of 12 inches of concrete.

(2) Process Samples Outside Work Stations

The maximum total mass of all process samples outside the work stations within the Purex Analytical Laboratory shall be 250 grams plus the mass in one sample. These may be stored and transported without restriction.

(3) Transport of Disposal Bottles

Only one disposal bottle shall be in transit at one time.

The distance between the disposal bottle and any other quantity of uranium-233 in excess of 250 grams shall exceed 20 inches center-to-center.

(4) Sample Storage Station

The cabinet shall be anchored to the wall to prevent tipping.

The shelves used for storing uranium-233 solutions (including process samples) shall be spaced at least 23 inches apart.

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*Includes the dumb waiter between the Purex Analytical Laboratory and the Sample Gallery.

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The maximum number of shelves utilized for storing uranium-233 solutions shall be three (3).

The maximum total volume of all solutions stored on each shelf shall be two (2) liters.

The maximum total mass of uranium-233 in all solutions (including process samples) on each shelf shall be 500 grams.

The Sample Storage Station shall be separated from any work station by 12 feet distance, edge-to-edge, or by neutron-shielding equivalent of 12 inches of concrete.

e. Limits: The Rest of the Purex Plant

(1) Process Samples

The maximum total mass of uranium-233 in all process samples in the rest of the Purex Plant shall be 250 grams plus the mass in one additional sample. These may be stored or transported without restriction.

(2) Disposal Bottles

During the storage and/or transportation of disposal bottles in the rest of the Purex Plant, they shall be considered to be product containers. Specifications for storage and transportation of product containers are given in Section 4.4.14.

f. Limits: Transfer of Solutions to Process Vessels

Process samples or laboratory solutions shall not be transferred to a process vessel unless it is known that the quantity of uranium-233 in the solution and the tank combined is less than the quantity specified for the tank.

g. Transportation of Process Samples

(1) Transportation of process samples between the Purex Plant and any other part of the Chemical Processing Division shall be limited to 500 grams of uranium-233 in a single vehicle.

(2) Transportation of process samples that contain less than 15 grams of uranium-233 (total for the shipment) between the Purex Plant and other Hanford contractors are not restricted by criticality safety consideration. Transportation of larger amounts requires the prior issuance of a nuclear safety specification.*

---

*P. E. Reed, "Criticality Safety of Fissile Materials in the Chemical Processing Division", ISO-270, April 1, 1966.

[redacted]

4.4.14 Authorizations

Prepared By: Jack B. Kendall Date: 8-6-65
Process Engineer, Separations Process Engineering

Issued By: Hal C. Rathvon Date: 8-6-65
Manager, Separations Process Engineering

Reviewed By: K. R. Ridgway Date: 8-10-65
Sr. Engineer, Technological and
Environmental Safety

Approved By: R. E. Tomlinson Date: 8-13-65
Manager, Research and Engineering

Accepted By: Date: 12/2/65
Manager, Purex Operation

[redacted]

Revision Approvals (4.4 CRITICAL MASS HAZARDS CONTROL SPECIFICATIONS)

Issued By: Manager Separations Process Eng.	Reviewed By: Sr.Engr. Nuclear Materials	Approved By: Manager Research & Engineering	Accepted By: Manager Purex Section	Pages Revised
H. C. Rathvon Date: 3/3/66	G. R. Kiel Date: 3-7-66	R. E. Tomlinson Date: 3-7-66	S G Smolen Date 3-23-66 3-18-66	Rev. 4.4.1-2, 3-1, 6-1 thru 9-1 & 12-1. Date: 3-2-66
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Date:	Date:	Date:	Date:	Date:
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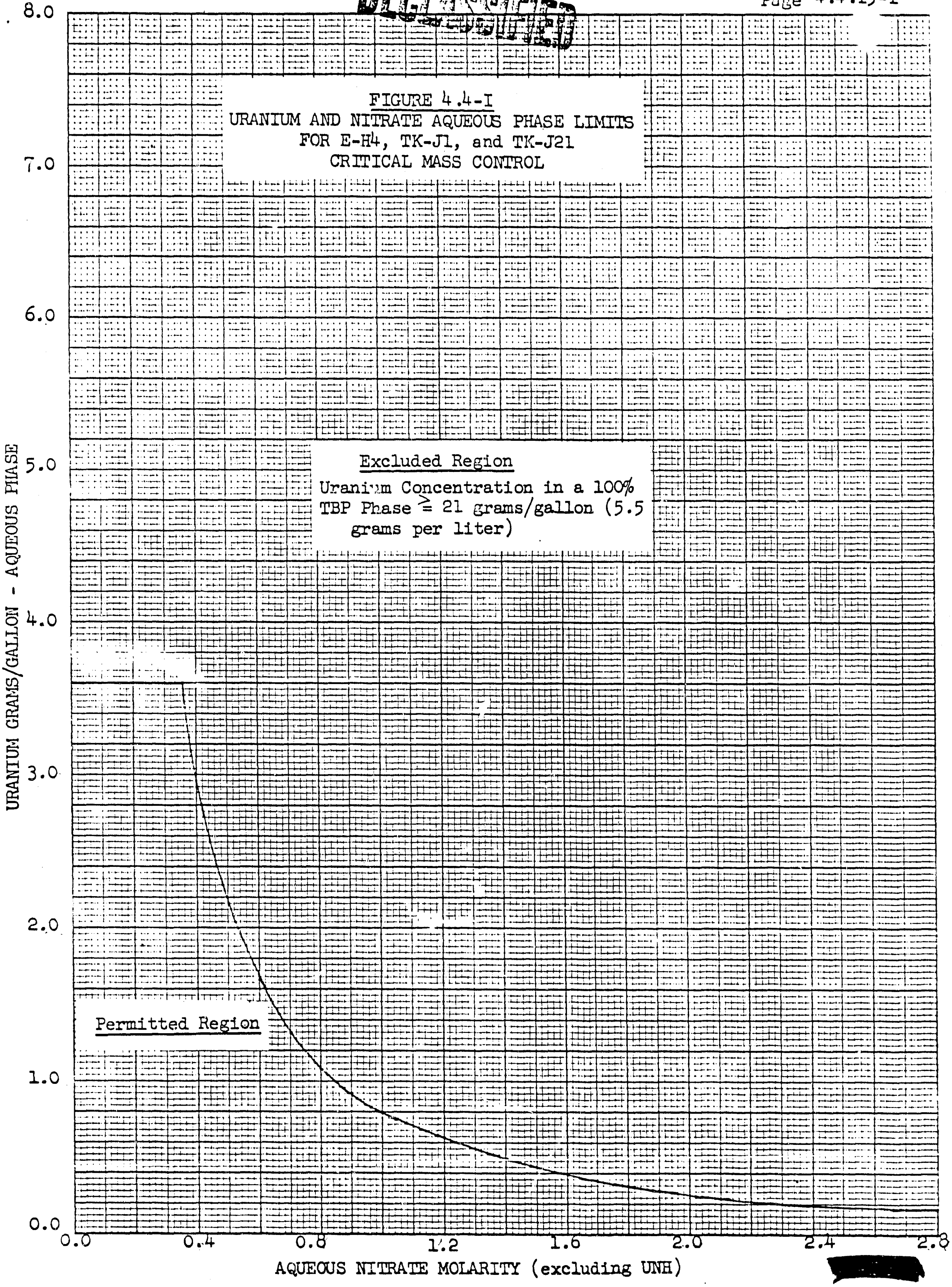

FIGURE 4.4-I
URANIUM AND NITRATE AQUEOUS PHASE LIMITS
FOR E-H4, TK-J1, and TK-J21
CRITICAL MASS CONTROL

[redacted]

[redacted]

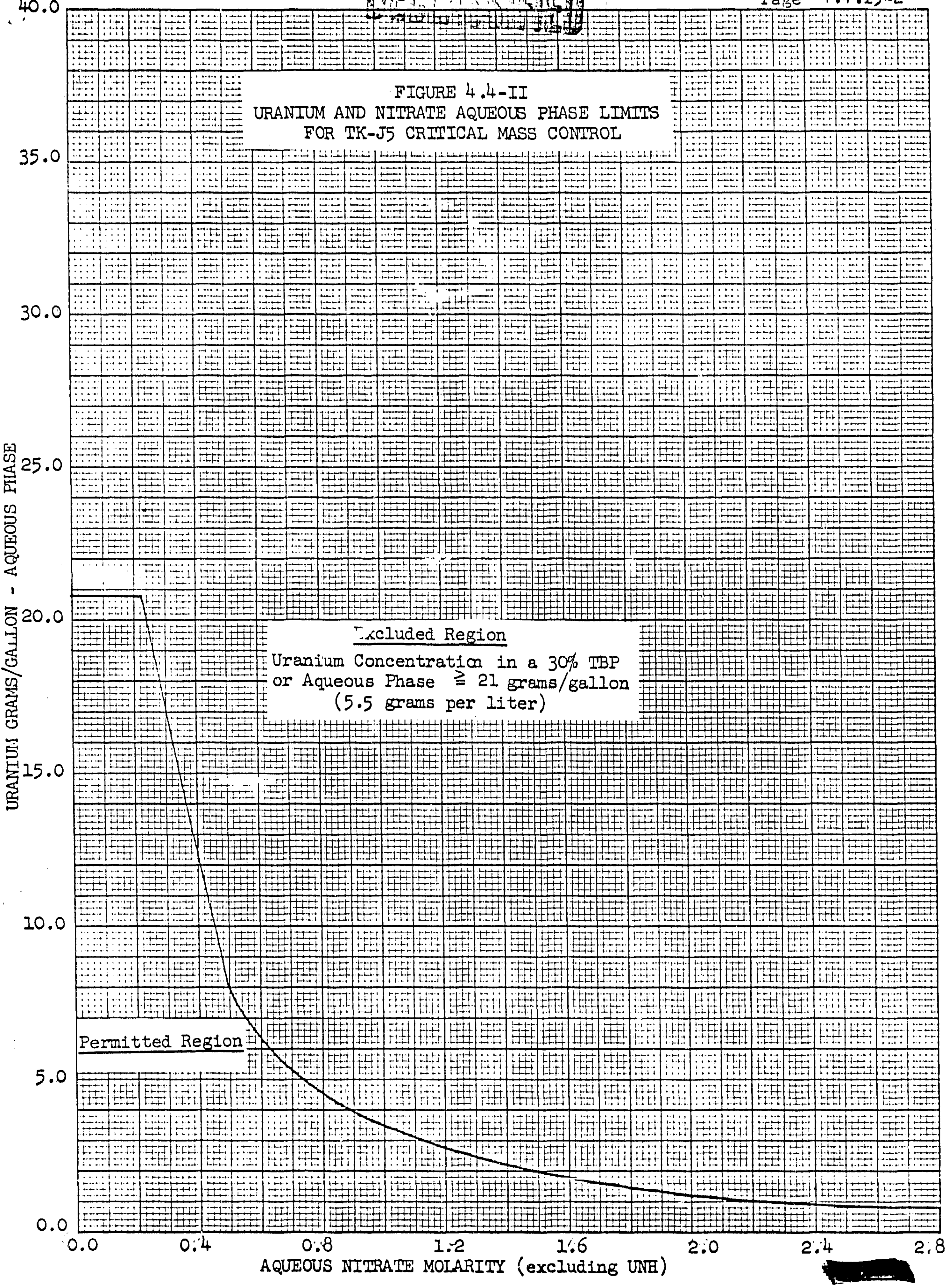

FIGURE 4.4-II
URANIUM AND NITRATE AQUEOUS PHASE LIMITS
FOR TK-J5 CRITICAL MASS CONTROL

[redacted]

[redacted]

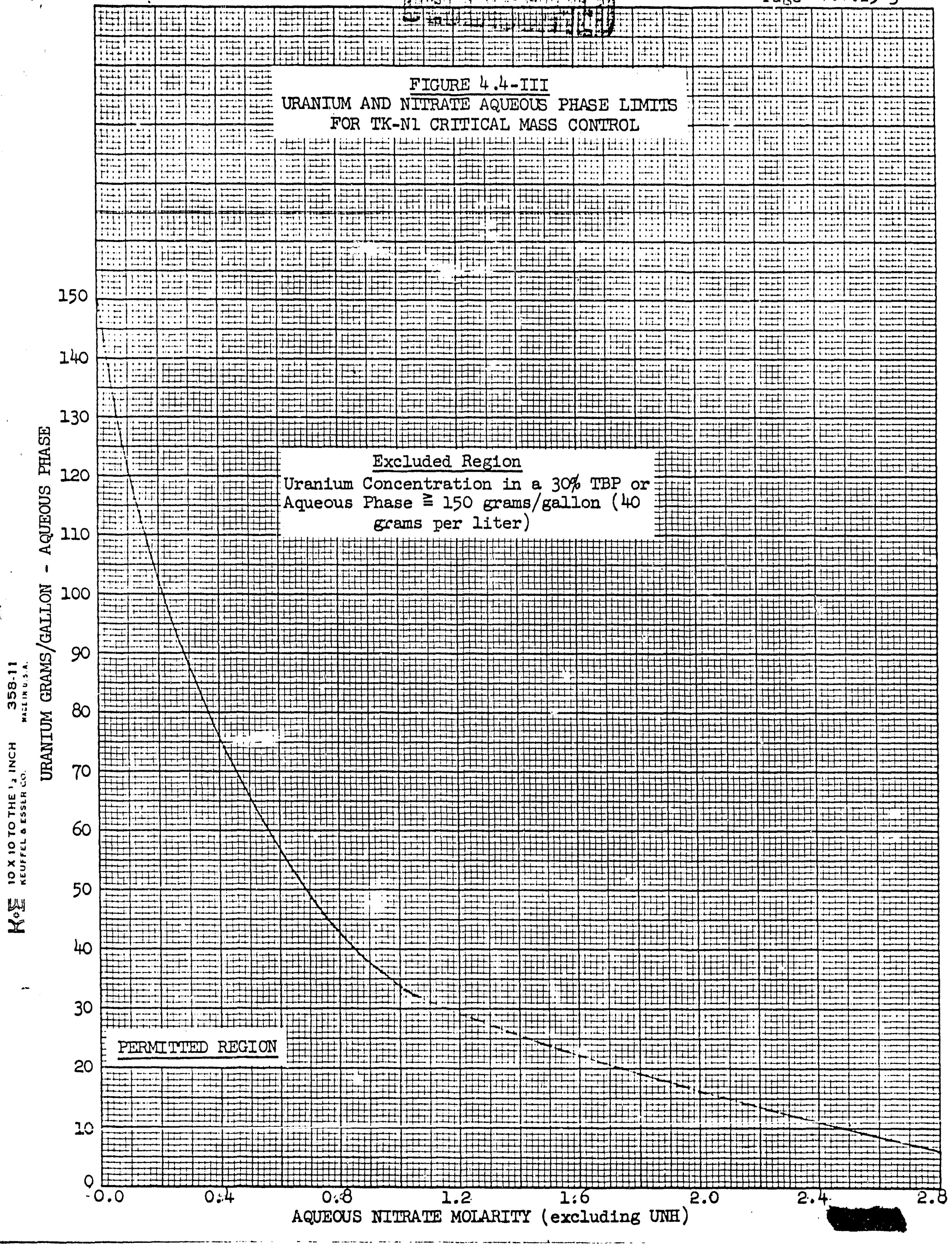

FIGURE 4.4-III
URANIUM AND NITRATE AQUEOUS PHASE LIMITS FOR TK-N1 CRITICAL MASS CONTROL

[redacted]

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H. C. Rathvon Date: 3/3/66	G. R. Kiel Date: 3-7-66	R. E. Tomlinson Date: 3-7-66	S G Smolen Date: 3-23-66	Added 4.4.12-2 & -3. and 4.4.14-2. Date: 3-2-66
H. C. Rathvon Date: 5/11/66	G. R. Kiel Date: 5-12-66	R. E. Tomlinson Date: 5-16-66	S G Smolen Date: 5-20-66	Rev. 4.4.2-1, 4.4.10-1, 4.4.11-1, and 4.4.12-2 & -3 Date: 5-6-66
H. C. Rathvon Date: 5/11/66	G. R. Kiel Date: 5-12-66	R. E. Tomlinson Date: 5-16-66	S G Smolen Date: 5-20-66	Renumbered 4.4.14-1 & -2 to 4.4.16-1 & -2. Added 4.4.14-1, 2 & 3. Date: 5-6-66
H. C. Rathvon Date: 5/11/66	G. R. Kiel Date: 5-12-66	5-16-66 Date: R. E. Tomlinson	S G Smolen Date: 5-20-66	Deleted 4.4.13-1 Date: 5-6-66
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Issued By: Manager Separations Process Eng.	Reviewed By: Sr.Engr. Nuclear Materials	Approved By: Manager Research & Engineering	Accepted By: Manager Purex Section	Pages Revised
H. C. Rathvon Date: 3/3/66	G. R. Kiel Date: 3-7-66	R. E. Tomlinson Date: 3-7-66	S H Smolen Date 3-23-66 3-18-66	Rev. 4.4.1-2, 3-1, 6-1 thru 9-1 & 12-1. Date: 3-2-66
H C Rathvon Date: 3/3/66	G. R. Kiel Date: 3-7-66	R. E. Tomlinson Date: 3-7-66	S H Smolen Date: 3-23-66 3-18-66	Added 4.4.12-2 & -3. and 4.4.14-2. Date: 3-2-66
H. C. Rathvon Date: 5/11/66	G. R. Kiel Date: 5-12-66	R. E. Tomlinson Date: 5-16-66	S H Smolen Date: 5-20-66	Rev. 4.4.2-1, 4.4.10-1, 4.4.11-1, and 4.4.12-2 & -3 Date: 5-6-66
H. C. Rathvon Date: 5/11/66	G. R. Kiel Date: 5-12-66	R. E. Tomlinson Date: 5-16-66	S H Smolen Date: 5-20-66	Renumbered 4.4.14-1 & -2 to 4.4.16-1 & -2. Added 4.4.14-1, 2 & 3. Date: 5-6-66
H. C. Rathvon Date: 5/11/66	G. R. Kiel Date: 5-12-66	5-16-66 Date: R. E. Tomlinson	S H Smolen Date: 5-20-66	Deleted 4.4.13-1 Date: 5-6-66
H. C. Rathvon Date: 5/27/66	G. R. Kiel Date: 5-27-66	R. E. Tomlinson Date: 5-27-66	S H Smolen Date: 5-27-66	Rev. 4.4.13-1 Added 4.4.13-2 & 4.4.13-3 Date: 5-27-66
Date:	Date:	Date:	Date:	Date:

Page Renumbered 5-6-66

4.5 Chemical Hazards Control Specifications

The chemicals that are used for thorium processing and the chemical characteristics of the thorium processing streams introduce potential hazards of fire, explosion, or other chemical reactions in the Purex Plant. The limits or restrictions necessary to maintain the probability of a hazardous incident at acceptably low levels are defined by the specifications below. They were developed in conjunction with the other specifications included in this document, and in conjunction with the chemical processes defined and recommended (Section 2.0) for processing the specified feed material (Section 4.1).

[redacted]

4.5.1 General

a. Additions and revisions to these specifications require the same level of approval as the specifications themselves.

b. Changes to the approved as-built thorium processing systems that could affect the chemical hazards considerations shall be approved by the Manager, Separations Process Engineering, prior to their processing use.

[redacted]

4.5.2 Radiolytic Heat

The quantity of radioactivity introduced to any process vessel shall be limited such that adequate cooling capacity is available for maintaining the vessel solution temperature at less than 50°C. See Section 3.10 for control guidelines.

[redacted]

[redacted]

4.5.3 Hydrogen Formation

a. Radiolytic Formation of Hydrogen

Vessel atmospheres shall be controlled by air dilution such that the calculated concentration of hydrogen gas formed by solution radiolysis does not exceed two volume per cent based on a hydrogen formation rate of 0.043 cubic feet per kilowatt-hour of radioisotopic energy. See Section 3.10 for control guidelines.

b. Chemical Formation of Hydrogen

Sodium hydroxide shall not be added to a dissolver charge of aluminum-clad thorium slugs unless the slugs are covered with enough sodium nitrate solution of at least 0.2 M concentration to give a mole ratio of sodium nitrate to aluminum (slug jacket) of at least 1.0.

[redacted]

4.5.4 Organic Vapors

a. For purposes of this specification, organic shall be considered to be normal paraffin hydrocarbon (NPH) diluent in any combination with tributyl phosphate (TBP).

b. For purposes of this specification any process stream that "might contain an organic phase" shall include any stream that has been previously in contact with an organic phase. Likewise, any vessel to which "an organic phase might be introduced" shall include any vessel that receives a stream that might contain an organic phase.

c. Any process vessel, including any condenser, to which an organic phase might be introduced, shall not be operated above 65° C, except as permitted by the conditions described by (1) and (2), below.

   (1) For vessels that are required to operate above 65° C, any influent liquid process stream that might contain an organic phase shall be pretreated for organic removal by decanting or by steam stripping. If steam stripping is used, the weight ratio of steam vapor to liquid shall be at least 0.5.

   (2) Vessels containing solutions that have contacted an organic phase and have not been steam stripped or decanted shall be operated above 65° C only under the conditions outlined in a special procedure provided by Purex Processing and approved by the Manager, Separations Process Engineering.

4.5.5 Solvent Nitration

The potential for solvent degradation or subsequent nitration reactions during the thorium run shall be minimized by compliance with the following:

a. Concentrator Systems

Introduction of Solvent: Process solutions shall be steam stripped prior to concentration in concentrators E-J8, E-K4, and E-H4. Effective control of the steam-stripping operation shall be maintained by (1) initially starting up the concentrators by establishing and maintaining the boiling of solvent-free solutions (such as water, fresh acid, or concentrator bottoms from previous operation), and (2) introduction of the process solutions to a steam stripping tower (T-J8, T-K4, T-H4). The feed solutions to concentrators E-F6 and E-N6 shall be pumped from unagitated tanks (TK-F12, TK-N1) in which the pump suctions are covered with aqueous solutions.

Vessel Pressure: The concentrator systems (E-J8, E-K4, E-H4, E-F6, E-N6) shall not be operated under positive pressure as measured by pot vacuum and differential pressure instrumentation. Accidental pressurization (positive) shall be immediately corrected.

Tube Bundle Surface Temperature: The concentrators shall be operated at or below the following maximum tube bundle steam pressures (and corresponding tube bundle surface temperatures) as measured by steam line pressure gauges:

E-K4, E-J8, E-H4, E-N6	29 psig (135° C)
E-F6	39 psig (141° C)

b. All Process Vessels

The maximum solution temperature in any vessel containing solution which has contacted a solvent phase shall be 125° C, as measured by in-vessel thermohms, except that for solutions that have not been steam stripped or decanted, a temperature of 65° C may be exceeded only under the conditions specified by Paragraph c (2), Section 4.5.4.

Note: The concentrators and appropriate tanks are equipped with high temperature alarms set at 120.5° C. If any vessel solution temperature exceeds 120.5° C, immediate action should be taken to keep the temperature below the 125° C maximum.

4.5.6 Ammonia Explosion Control

To prevent the formation of explosive ammonia conditions in the Purex Plant during the run, the following specifications are required:

a. Dissolver System

(1) During the coating removal operation, the unheated dissolver off-gas temperature for any dissolver system shall be maintained above 45° C as measured by a thermohm in the gas outlet from the downdraft condenser. Also, during coating removal, the ammonia scrubber water rate shall be a minimum of 10 gallons per minute.

(2) The silver reactor influent gas shall be maintained between 350 and 400°F during coating removal, dissolution, and boiling for denitration.

b. Waste Neutralization

The minimum solution temperature during waste neutralization in vessels TK-F15, TK-F16, TK-F18, TK-R8, and TK-G8 shall be 45° C as measured by in-vessel thermohms. Note: See Section 4.5.4 for maximum operating temperatures.

[redacted]

4.5.7 Fluoride Corrosion Control

Fluoride-induced corrosion of stainless-steel equipment in the Purex Plant will be minimized during the thorium run by adding aluminum nitrate nonohydrate (ANN) at a mole ratio equal to or greater than 4 moles of ANN for each mole F^- to any vessel which has a fluoride concentration of > 0.001 M.

[redacted]

4.5.8 Waste Neutralization

Wastes transferred from the Purex Plant to any underground waste storage tank shall first be neutralized to a pH of 10 or greater, except that solvent-wash wastes containing sodium carbonate require no additional neutralization if the pH is 8, or greater.

[redacted]

4.5.9 Authorizations

Prepared By: S M Nielson Date: 9/1/65
Process Engineer, Separations Process Engineering

Issued By: H. C. Rathvon Date: 9/1/65
Manager, Separations Process Engineering

Approved By: R. E. Tomlinson Date: 10-8-65
Manager, Research and Engineering

3-3
Accepted By: S G Smolen Date: 3-3-66
Manager, Purex Operation

[redacted]

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DATE FILMED

3/4/92