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 Concentrating Second Uranium
 Cycle Product
 TO: F. L. Culler
 FROM: W. L. Albrecht

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AEC RESEARCH AND DEVELOPMENT REPORT

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1.0 INTRODUCTION

In the ORNL Thorex Process, uranium-233 is separated from thorium, protactinium and fission products by solvent extraction and is further decontaminated and concentrated by an ion-exchange process. The uranium obtained by this method during a series of production runs in 1956 was insufficiently decontaminated from fission products to permit further processing at another site. The activity was further decreased by processing the ion-exchange product through an additional solvent-extraction cycle in which glass columns were employed. The uranyl nitrate product solution from the second cycle was of such a concentration that it was desirable to reduce its volume prior to shipment. A system for concentrating this solution by evaporation was designed and installed in the Thorex Pilot Plant.

The concentration system includes a feed tank, an evaporator, and a condensate catch tank. The evaporator has a steam-jacketed falling film section and a receiver section. Parts of the evaporator that come in contact with solution are made of tantalum to minimize corrosion. Batches of solution, limited by criticality considerations to 300 g uranium-233, are continuously fed to the evaporator at a rate of 150 ml/min during a run.

The concentration system was operated from May 14 to July 31, 1956 to concentrate second-cycle uranyl nitrate product to reduce the volume of solution to be shipped. The uranium solution concentrated was a portion of that separated in Thorex Runs HD-16 through HD-18. Experience gained in operating the system indicated that certain minor modifications would improve operation of the batch system, and also suggested that the evaporator might be adapted to continuous operation.

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2.0 SUMMARY

A total of 16,060 g of uranium was concentrated in the system in 68 batch runs. A total of 14,400 g total uranium (14,180 g U-233) was recovered as product suitable for shipment. Uranium loss to the evaporator condensate was 0.03 per cent of the total uranium processed. The material balance across the system was 98.4 per cent.

The average concentration of uranium in the evaporator feed solution was 29 g/liter; the average concentration in the evaporated solution was 298 g U/liter and in the product solution was 199 g/liter. Radiation readings of bottles containing product solutions were taken with a hard-shell cutie pie immediately after each run, and these readings ranged from 35 to 1100 mr/hr. The radiation levels of the bottles of product solution shipped averaged 78 mr/hr. Bottles of product solution reading in excess of 300 mr/hr, maximum allowable for shipment, were reprocessed in the second-cycle solvent extraction system and reconcentrated. The products from seven runs had radiation levels in excess of 300 mr/hr at the time of concentration, or the activities had grown to that level by the time of shipment.

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3.0 DESCRIPTION OF THE CONCENTRATION SYSTEM

3.1 Equipment

The concentration system (Figure 3-1) is located in a hood. The major items of equipment are the feed tank (L-20), the batch evaporator (L-21), the condenser (L-22), and the condensate tank (L-23). Feed for the system is delivered from the second solvent-extraction system in 4-liter polyethylene bottles (PB). Service piping for demineralized water, process water, vacuum, air, and radioactive off-gas is located behind the hood. A steam line enters the hood at the top, above the evaporator.

The evaporator (Figure 3-2) is a batch-type, steam-jacketed, falling-film evaporator. All parts of the evaporator that come in contact with solution or vapor are made of tantalum. Other parts are made of type 347 stainless steel.

The falling-film section of the evaporator is a tantalum tube 2 in. OD by 0.03 in. thick by 31 in. long. The solution is distributed at the top of the tube so that it passes down the tube wall in a thin film. The receiver section is a tantalum pot 4 in. OD by 0.04 in. thick by about 11 in. deep. A small depression is provided at the bottom of the pot to permit substantially complete removal of product through the product removal probe. Circumferential grooves lend rigidity to the thin-walled pot. The capacity of the product receiver is about 2 liters. At the level of the upper condensate from the jacket drain, the usual operating level, the receiver contains about 1 liter of solution.

The feed tank and the condensate are each made of 5 in. IPS, 54 in. long, schedule 40 stainless steel pipe; each has a capacity of about 17.4 liters. Each tank is fitted with a 3/8 in. heavy wall Tygon tubing sight glass.

The water-cooled condenser is a jacketed 3/4 in. IPS schedule 40 stainless steel pipe between the evaporator and the condensate tank.

Stainless steel caps mounted on a rack are used to support the feed and product bottles. The caps are threaded to match the threads on the 2- and 4-liter polyethylene bottles.

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Fig. 3-1. CONCENTRATION SYSTEM

Fig. 3-2
CROSS SECTION OF
EVAPORATOR

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3.2 Instrumentation

The feed tank is equipped with a sight glass for measuring liquid level in the tank. A rotameter (range 0 to 150 ml/min) in the feed line to the evaporator is used for indicating the rate of flow of solution from the feed tank to the evaporator. A manometer indicates the level of solution in the receiver section of the evaporator. The condensate tank has a sight glass for measuring liquid level in the tank. Vacuum in the product bottle is measured with a guage.

The manometer gives only an approximate indication of the volume of solution in the receiver section of the evaporator because the specific gravity of the solution is not known, and the manometer reading fluctuates considerably on account of boiling in the receiver. The volume of solution in the receiver is calculated more accurately by taking the difference between the volume depletion in the feed tank and the volume build-up in the condensate tank.

3.3 Criticality

In order to avoid any possibility of creating a critical mass, individual batches are limited to a maximum of 300 grams U-233.

3.4 Operation of the System

The concentration system is operated batchwise (see Figure 3-3 for flow diagram). A feed bottle is screwed onto the rack-mounted feed cap, and the contents are transferred to the feed tank by applying vacuum to the feed tank. The contents of two or three bottles, plus about 0.5 liter of rinse water from each, are transferred in this manner. Thereafter, air pressure (about 5 psig) is applied to the feed tank, and approximately 1 liter of solution is transferred to the evaporator, where it is collected in the receiver section.

Next, the condenser cooling water is turned on, and about 10 psig steam is admitted to the jacket of the evaporator. About 3 min is required to bring the evaporator to operating temperature. When the evaporator is hot, the feed solution is transferred by pressure to the evaporator at a rate of about 150 ml/min.

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Fig. 3-3
FLOW DIAGRAM OF CONCENTRATION
SYSTEM

The evaporation usually progresses smoothly, with no adjustment required other than regulation of the feed rate. It may be necessary to increase or decrease the steam pressure in the evaporator jackets so as to increase or decrease the boil-up rate, but such adjustment is required only infrequently. Usually, the volume of solution in the evaporator receiver section remains fairly constant throughout the run within the range of about 0.8 to 1.1 liters.

When the feed tank is empty, as evidenced by the appearance of air in the rotameter, the steam is turned off, and boiling of the solution in the receiver section is stopped by admitting water to the lower jacket of the evaporator.

After the solution in the product receiver has been cooled for about 15 min, it is transferred to the product bottle by applying vacuum to the product bottle.

After each run in a series,^a the feed tank and evaporator are flushed by adding about 0.3 to 0.5 liter of demineralized water to the feed tank and successively transferring the water to the evaporator and the product bottle. After each series of runs, the feed tank and evaporator are flushed by adding about 2 liters of demineralized water to the feed tank and evaporating the water to about 0.5 liter. The resulting solution is transferred to the product bottle.

The product is monitored with hard-shell and paper-shell cutie pie radiation meters before it is placed in a "bird-cage" shipping container.

After each run, a sample of the condensate is taken. The condensate usually contains approximately 0.01 g/liter of U-233, based on gross alpha counting. If the total U-233 content of the condensate is less than about 0.2 g, the condensate is discarded to waste.

a. The solution produced in a single run of the second solvent-extraction system furnishes feed for a "series" of concentrator runs.

4.0 OPERATING RESULTS

Pertinent data from the first 68 runs made in the concentration system are shown in Table 4-1.

A total of 16,060.27 g of uranium was processed in 68 runs of the concentration system. Of this amount, 14,399.91 g was shipped, and 1,660.36 g was recycled to the second solvent-extraction system.

The input to the system was 16,322.8 g of uranium; 98.4 per cent of the uranium was accounted for.

The average uranium content of the feed solution was 29 g/liter. The concentration of the feed varied widely due to the variation in concentration of the second-cycle product and to variation in the amount of water used to flush the feed bottles. In some runs, the feed was diluted by the accumulated heel from feed bottles used in previous runs; the heel from each bottle amounted to about 0.1 liter.

The solution collected in the receiver section of the evaporator had an average concentration of 298 g U/liter. The average concentration factor was 10.4. The final product, which was diluted by water used to flush the evaporator, had an average uranium content of 199 g/liter.

The loss of U-233 (measured by gross alpha counting) in the condensate amounted to 0.03 per cent of the total uranium processed in the concentration system. Approximately 78 per cent of the loss occurred in Run 29, when the loss amounted to 1.75 per cent. An unsuccessful attempt was made to recover the condensate in this run. The average loss, not including Run 29, was 0.0072 per cent.

Following the last run (number 68), the feed tank and evaporator were cleaned out by filling the feed tank with 2N nitric acid and boiling the acid in the evaporator. A total of 0.27 g U was collected in 4.2 liters of acid; the acid was discarded to waste.

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Table 4-1

Operating Results of Concentration System

Key to column headings:

A = Run number	G = Concentration of final product,
g total U/liter
B = Product bottle number	H = U-233 loss in condensate, grams
C = Total uranium in product, grams	J = U-233 loss in condensate, % of total
U processed
D = Concentration of evaporator feed,
g total U/liter	K = Product radiation levels, mr/hr
E = Concentration of evaporated solution,
g total U/liter	K-1 = Hard-shell cutie-pie
F = Concentration factor = E/D	K-2 = Paper-shell cutie-pie

A	B	C	D	E	F	G	H ^a	J	K	
									K-1	K-2
1	88	269.50	31	290	9.4	181	0.0021	0.0008	68	90
2	89	294.62	24	222	9.3	183	0.0028	0.0010	87	120
3	90	258.11	24	385	16.0	173	0.0050	0.0019	65	95
4	91	283.94	23	308	13.4	260	0.0064	0.0023	78	100
5	92	260.48	23	302	13.1	166	0.0062	0.0024	70	100
6	93	255.53	14	138	9.9	117	0.0004	0.0004	70	86
7	94	252.27	22	287	13.0	175	0.0096	0.0038	70	88
8	95	249.63	33	268	8.1	159	0.0098	0.0039	62	78
9	96	247.15	29	309	10.7	176	0.0055	0.0022	67	85
10	84	255.53	33	335	10.1	212	0.0041	0.0015	70	95
11	85	255.53	30	229	7.6	151	0.0020	0.0009	76	100
12	97	255.53	31	299	9.6	183	0.0077	0.0027	70	100
13	107	247.99	26	243	9.4	165	0.0042	0.0017	55	75
14	108	291.20	38	300	7.9	185	0.0081	0.0028	60	80
15	110	266.86	37	267	7.2	206	0.0037	0.0014	60	75
16	111	216.53	22	555	25.2	168	0.0050	0.0023	90	120
17	77	195.62	20	194	9.7	132	0.0025	0.0013	80	110
18	78	285.45	38	304	8.0	197	0.0038	0.0013	60	85
19	87	291.75	38	435	11.5	214	0.0150	0.0035	55	90
20	116	136.44	23	83	3.9	94	0.0150	0.0035	45	70
21	117	255.93	31	388	12.5	229	0.0060	0.0023	70	85
22	118	241.90	28	361	12.9	229	0.0114	0.0047	55	80
23	119	237.41	28	258	9.2	182	0.0064	0.0027	65	85
24	120	249.72 ^c	27	595	22.0	174	0.0162	0.0065	85 ^b	110 ^b
25	121	195.18 ^c	27	246	9.1	139	0.0181	0.0093	90 ^b	110 ^b
26	122	215.19	29	245	8.5	182	0.0050	0.0023	75	100
27	123	201.46	25	219	8.8	176	0.0058	0.0029	65	85
28	124	222.43 ^c	26	304	11.7	182	0.0053	0.0024	78 ^b	110 ^b
29	125	210.28 ^c	26	239	9.2	149	3.675	1.748	90 ^b	110 ^b
30	126	224.44	26	218	8.4	171	0.299	0.1333	190	270
31	127	218.10	27	190	7.0	151	0.0427	0.0196	200	270
32	128	228.71	29	276	9.5	153	0.0127	0.0055	150 ^b	200 ^b
33	133	208.40 ^c	26	278	10.7	-	0.0090	0.0043	380 ^b	500 ^b
34	134	265.32 ^c	30	282	9.4	-	0.0149	0.0056	170 ^b	220 ^b

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Table 4-1 (Cont'd)
Operating Results of Concentration System

A	B	C	D	E	F	G	H	J	K	
									K-1	K-2
35	143	295.95 ^c	21	274	13.0	237	0.0299	0.0101	750 ^b	975 ^b
36	144	257.83	33	277	8.4	204	.0072	0.0028	130	240
37	145	273.33	33	342	10.4	202	.0053	0.0019	110	190
38	151	235.58	30	274	9.1	206	.0098	0.0042	73	80
39	152	233.03	30	324	10.8	203	.0087	0.0037	50	60
40	153	241.58	27	278	10.3	218	.0094	0.0039	62	100
41	157	226.26 ^c	19	269	14.1	-	.0417	0.0184	1100 ^b	2000 ^b
42	158	258.97 ^c	23	328	14.2	-	.0408	0.0158	750 ^b	1000 ^b
43	159	222.74	28	268	9.6	209	.0114	0.0051	65	85
44	160	243.03	32	324	10.1	254	.0084	0.0035	62	80
45	161	246.46	29	297	10.2	165	.0111	0.0045	62	80
46	163	226.83	28	244	8.7	246	.0073	0.0032	78	100
47	99	260.92	32	256	8.0	198	.0027	0.0010	70	100
48	162	267.33	32	294	9.2	227	.0067	0.0025	75	95
49	154	154.17	25	328	13.1	139	.0037	0.0024	70	95
50	155	234.47	31	321	10.4	161	.0065	0.0028	80	100
51	164	255.37	33	364	11.0	283	.0071	0.0028	78	90
52	165	246.10	32	212	9.8	238	.0084	0.0034	65	100
53	166	251.40	22	364	16.5	236	.0214	0.0085	80	120
54	167	227.00	28	234	8.4	200	.1250	0.0550	110	130
55	168	103.27	11	344	31.2	133	.0036	0.0035	35	60
56	169	258.14	32	208	6.5	237	.0027	0.0011	90	120
57	170	288.34	35	267	7.6	271	.0266	0.0092	83	100
58	171	267.27	33	257	7.8	260	.0147	0.0055	78	95
59	172	144.08	23	236	10.3	194	.0122	0.0085	62	74
60	173	248.60	31	251	8.1	235	.0131	0.0053	80	100
61	174	280.78	34	278	8.2	245	.0060	0.0021	65	100
62	175	278.40	35	278	7.9	243	.0065	0.0023	-	100
63	176	110.73	29	217	7.5	135	.0044	0.0040	-	95
64	177	235.67	29	243	8.4	223	.0322	0.0137	100	100
65	178	244.72	30	245	8.2	263	.0467	0.0191	70	100
66	179	255.81	24	261	10.9	184	.0376	0.0147	74	98
67	180	180.59	25	188	7.5	178	.0051	0.0028	90	130
68	181	126.84	18	282	15.7	174	.0075	0.0059	55	76
Total	-	16,060.27	-	-	-	-	4.8228	-	-	-
Average	-	-	29	298	10.4	199	-	0.0301	78	105

- a. U-233 in condensate determined by gross alpha counting.
b. Not included in average.
c. Solution recycled to second solvent-extraction cycle. Total uranium recycled = 1,660.36 grams.

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5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The concentration system served its intended purpose very well, and was operated with a minimum of difficulty.

A drawback of the system is that the constant attention of an operator is needed while the system is in operation.

The placement of feed bottles on and the removal of product bottles from the rack-mounted caps is a rather awkward procedure. The likelihood that solution will ever be spilled in such handling probably is remote. Nonetheless, in view of the high value of the solutions, the risk of a spill is quite great.

5.2 Recommendations

The following modifications probably would permit four runs to be made per shift. With the concentration system in its present form, the maximum number of runs that can be carried out consistently during an 8-hour shift is three.

1. Installation of a cooler in the line from the evaporator receiver section to the product bottle. This would permit product solution to be transferred to polyethylene bottles immediately after an evaporation was completed. With the present system, the solution must be held in the receiver section for about 15 to 20 min before the solution is cool enough to be transferred to a product bottle.

2. Installation of a storage tank to which condensate could be transferred after each run. Condensate could be sent to the storage tank immediately after sampling, thus eliminating the time now lost while waiting for analytical results. Air pressure could be applied to the condensate tank to transfer the condensate to the storage tank. The condensate, which contains a small amount of uranium, could be used, for example, in the Thorex feed adjustment step.

If desired, the evaporator could be modified for continuous, remote operation. A proposed arrangement for such an operation, to be used in conjunction with the proposed third uranium cycle, is shown in Figure 5-1.

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Fig. 5-1

CONTINUOUS EVAPORATOR
for
SECOND CYCLE URANIUM PRODUCT

The equipment could be located in cell 3, with control instruments located on the third uranium cycle control panel. Since the capacity of the concentration system would exceed that of the third-cycle system, the evaporation system could be operated on a part-time basis, or the evaporator feed could be diluted. Operation of the system would be controlled by varying the feed rate and jacket steam pressure to maintain a desired product concentration.

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6.0 APPENDIX

6.1 Instructions for Operating Uranium Concentration System

The concentration system was installed early in May, 1956, to evaporate uranyl nitrate solution from a second uranium cycle solvent extraction system. The second-cycle system operated on solution eluted from the resin columns in the Thorex isolation laboratory.

Equipment for the concentration system is installed in a hood in the East Dock area of Building 3019.

Detailed instructions and discussions of operating techniques are given in this section.

6.1.2 Symbols and Abbreviations

Symbols and abbreviations used in these instructions are given below:

1. Equipment

L-20 - Feed tank
L-21 - Evaporator
L-22 - Condenser
L-23 - Condensate catch tank
FB - Feed bottle
PB - Product bottle

2. Service Lines

PW - Process water line
DW - Demineralized water line
VAC - Vacuum line
AIR - Air line
VOG - Vessel off-gas line
ST - Steam line
DN - Drain line

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3. Instruments

- FI-L-20 Flow indicator (rotameter) in line from L-20 to L-21
- PI-L-20 Pressure indicator, showing air pressure in L-20
- PI-L-21 Pressure indicator, showing steam pressure in L-21 jacket
- LI-L-21 Liquid level indicator (manometer), showing level of solution in the receiver section of L-21
- PI-PB Pressure indicator, showing vacuum in the product bottle

4. Valves

- PV-L-21 Pressure regulating valve for steam supply to L-21 jacket
- PV-L-20 Pressure regulating valve for air supply to L-20

Valves in process lines are designated by a 3-symbol system (e.g., V-L20-L21) in which the first symbol indicates a valve, the second indicates the source of the stream, and the third indicates the destination of the stream. Thus, V-L20-L21 is the designation for the valve in the feed line from L-20 to L-21. The symbol V-PB-VAC designates the valve in the line from the product bottle to the vacuum line.

6.2 Instructions for Operating the Uranium Concentration System

6.2.1 Outline of the Process

A flow diagram of the process is shown in Figure 3-3.

In general, the process consists of the following steps:

- a. Solution is transferred from the feed bottles to the feed tank.
- b. Solution is fed from the feed tank to the evaporator, where it is concentrated to a volume of about 1 liter.
- c. The concentrated product solution is cooled and transferred to the product bottle.
- d. The feed tank and evaporator are flushed with demineralized water and the wash solution is transferred to the product bottle. In the last run of a series of runs, a boil down wash is carried out and the solution is added to the product bottle.

- e. The condensate catch tank is sampled and the contents, if sufficiently low in uranium, are discarded to waste.
- f. The product bottle is removed, monitored, and placed in a bird cage.

6.2.2 Description of Equipment

- a. Feed bottles are 4-liter polyethylene bottles.
- b. Product bottles are 2-liter polyethylene bottles.
- c. The feed tank is a vertically-mounted, cylindrical tank of welded construction 54 in. long. The tank is made of 5-in., IPS, schedule 40, stainless steel pipe and has flat heads on top and bottom. Nozzles are provided at the top for feed, demineralized water, vacuum, air, and off-gas connections. An outlet to L-21 is provided at the bottom. The tank is fitted with a sight glass calibrated in liters.
- d. A cross sectional view of the evaporator is shown in Figure 3-2.
- e. The condenser consists of a water-cooled jacket around the 3/4 in., IPS schedule 40 pipe between L-21 and L-23. The jacket is made of stainless steel tubing (1-1/4 in. OD, 0.065 in. wall, 48 in. long).
- f. The condensate catch tank is identical to L-20 except for the nozzles in the top plate. L-23 has nozzles for condensate, off-gas, and air. The outlet at the bottom leads to a sample point and to the drain.

6.2.3 General Operating Rules

The following safety rules should be observed:

- a. Do not exceed 300 g of uranium per run.
- b. Do not place full feed bottle near full product bottle.
- c. Keep full product bottles in bird cages.
- d. Do not open L-20 sight glass valves while air pressure is on L-20. First, close V-AIR-L20; second, open V-L20-VOG.
- e. Keep V-L23-VOG open at all times, except when transferring solution from L-21 to PB and when draining L-23.

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In order to minimize personal contamination and general contamination of the area, the following procedures should be followed:

- a. Handle feed bottles with rubber gloves only, and use these gloves for that purpose only.
- b. Place rubber gloves inside the hood on blotter paper when not in use.
- c. Wear cloth gloves when handling valves or extension handles located outside the hood.
- d. Handle valves inside the hood with built-in gloves or with a sheet of disposable tissue.
- e. Use disposable tissue to open and close hood doors.
- f. Handle bird cages with cloth gloves.
- g. Place clean polyethylene bag on product bottle before removing the bottle from the rack.

6.2.4 Operating Procedure

6.2.4.1. Transfer of Feed to L-20

Transfer of solution from FB to L-20 is accomplished by applying a vacuum to L-20 and allowing atmospheric pressure to force solution from FB to L-20. The operating sequence is as follows:

- a. Place FB on the stainless steel cap.
- b. Close V-L20-VOG.
- c. Open V-L20-VAC about one and one-half turns.
- d. Open V-FB-L20.
- e. When FB is empty, close V-FB-L20.
- f. Open V-DW-FB, add about 0.5 liter of water to FB.
- g. Repeat steps d and e.
- h. Remove FB from cap, insert stopper, screw on cap, and set aside for cleaning.
- i. Repeat the foregoing procedures for second and third feed bottles.
- j. Close V-L20-VAC; open V-L20-VOG.

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The maximum amount of uranium-233 to be processed in a given run is 300 g. Most runs are made concentrating the contents of two feed bottles, although three bottles may be handled if the total uranium-233 content does not exceed 300 g.

6.2.4.2. Evaporation of Solution

Evaporation of the solution is started with about 1 liter of solution in the receiver section of L-21. The operating sequence for transferring this amount of solution to L-21 is as follows:

- a. Record liquid level in L-20.
- b. Close V-L20-VOG.
- c. Open V-AIR-L20; adjust PV-L20 to 5 psig on FI-L20.
- d. Open V-L20-L21 and adjust to a flow rate of 150 ml/min as indicated on FI-L20. Maintain a flow rate of 150 ml/min for 6.5 min, then close V-L20-L21.
- e. Close V-AIR-L20; open V-L20-VOG.
- f. Record L-20 liquid level.

With approximately 1 liter of solution in the L-21 product receiver and with L-23 empty, the evaporation is carried out as follows:

- a. Open V-PW-L22 about 1/4 turn to admit cooling water to L-22.
- b. Open V-ST-L21 to admit steam to L-21 jacket; adjust PV-L21 to 8 to 10 psig.
- c. Close V-L20-VOG; open V-AIR-L20. When condensate begins flowing through the Tygon tube connecting L-22 and L-23, or when 3 min have elapsed after opening V-ST-L21, open V-L20-L21; adjust flow to 150 ml/min on FI-L20.
- d. Record L-23 level and LI-L-21 readings at 10 min intervals after V-L20-L21 is opened.

The flow of solution from L-20 to L-21 is maintained at 150 ml/min until L-20 is empty. The flow rate varies, and should be readjusted every few minutes. The appearance of air bubbles in FI-L-20 indicates that L-20 is empty. V-L20-L21 should be closed as soon as air bubbles appear to avoid possible carry over of solution to L-23.

During the evaporation, it is desirable to maintain the volume of solution in L-21 receiver at about 1 liter. There is no need to take steps to control this volume, however, unless it exceeds about 1.8 liters or is less than about 0.5 liter. The volume in L-21 receiver is indicated by LI-L-21, but because the specific gravity of the solution is not known; LI-L-21 gives only a rough approximation of the volume. The volume can be determined quite accurately by the difference between the depletion in L-20 and the build-up in L-23.

The evaporation normally requires no control other than periodic regulation of the solution flow rate. The following control measures can be taken if necessary:

- a. If the volume in L-21 receiver increases to about 1.8 liters, the solution flow should be stopped until the solution in the receiver is boiled down to about 1 liter. The boil-up rate in the receiver can be increased by closing the valve in the condensate by pass line.
- b. If the volume in L-21 receiver decreases to about 0.5 liters, the steam should be cut off until the volume builds up to about 0.8 liters.

The volume in L-21 receiver at the end of the evaporation should be between about 0.6 and 1.1 liters. If, as the end of the evaporation approaches, it appears that the volume is less than 0.6 liters, the steam should be cut off before L-20 is empty. If the volume is more than 1.1 liters when L-20 is empty, the steam should be left on until the volume is less than 1.1 liters.

After V-ST-L21 and V-L20-L21 have been closed, the contents of L-21 receiver are cooled by admitting water to the jacket via V-FW-L21. The water should be admitted in short bursts; if PI-L-21 indicates a pressure rise, V-FW-L21 should be shut off. Cooling may be discontinued when the lower jacket of L-21 is cool to the touch.

V-AIR-L20 may be closed and V-L20-VOG opened at any convenient time after V-L20-L21 is closed. V-FW-L22 may be closed after V-ST-L21 is closed and condensate stops flowing from L-22 to L-23.

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6.2.4.3. Transfer of Solution to the Product Bottle

After the concentrated solution in L-21 receiver has been cooled, it is transferred to a 2-liter polyethylene bottle in accordance with the following procedure:

- a. Screw a 2-liter bottle to the stainless steel cap.
- b. Close V-L23-VOG; open V-L21-PB.
- c. Open V-PB-VAC carefully. Do not exceed about 2.5 in water vacuum, as indicated on PI-PB. When the needle on PI-PB drops quickly to zero, indicating L-21 receiver is empty, close V-PB-VAC and V-L21-PB; and open V-L23-VOG.

It is very important that V-L23-VOG be opened as soon as the transfer of solution to the product bottle is completed. Air for LI-L-21 entering the system can pressurize L-21, L-22 and L-23 sufficiently to cause damage if V-L23-VOG is kept closed for more than 5 to 10 min.

6.2.4.4. System Clean-out

After each run, L-20 and L-21 are flushed with a small amount of demineralized water (0.3 to 0.5 liter) which is added to L-20 via V-DW-L20. After the last of a series of runs, a boil-down wash is carried out instead of flushing with water. The boil-down wash is carried out as follows:

- a. Add about 2.0 liters of demineralized water to L-20 via V-DW-L20.
- b. Open V-AIR-L20; close V-L20-VOG.
- c. Open V-L20-L21; adjust flow rate to 150 ml/min.
- d. Open V-FW-L22.
- e. Within 4 to 5 min after V-L20-L21 is opened, open V-ST-L21.
- f. Boil down solution to a final volume of 0.3 to 0.5 liter in L-21 receiver, following procedure outlined in section 6.2.4, step 2.
- g. Cool solution in L-21 receiver and transfer to PB, following procedure outlined in section 6.2.4, step 3.

Flushing of L-20 and L-21 is carried out as follows:

- a. Add 0.3 to 0.5 liter of demineralized water to L-20 via V-DW-L20.
- b. Transfer water from L-20 to L-21 without heating.
- c. Transfer solution from L-21 receiver to PB.

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6.2.4.5. Sampling and Draining L-23

L-23 is sampled to determine the uranium content of the condensate. Sampling is carried out as follows:

- a. After checking to see that V-L23-VOG is open, open V-AIR-L23 a crack. Sparge for 5 min or more.
- b. Open V-L23-DR for about 10 seconds.
- c. Collect sample in 1-oz. polyethylene bottle. Code L-23-oo-1. Request gross α (estimate = $1 \times 10^4 \alpha$ cts/min/ml).

If analysis indicates that the uranium content of the condensate in L-23 is less than about 0.2 g, close V-L23-VOG; open V-L23-DR. Run until L-23 is empty, then close V-L23-DR; open V-L23-VOG.

If the total amount of U-233 in L-23 exceeds about 0.2 g, transfer solution to L-20 by the following procedure:

- a. Close V-L23-VOG. Open every few minutes to relieve pressure.
- b. Empty L-23 by draining into a polyethylene beaker via the sampling valve. Pour contents of beaker into 4-liter polyethylene bottles.
- c. Process the solution in subsequent evaporation runs.

6.2.4.6. Handling of the Product Bottle

The following procedure is followed in handling the product bottle:

- a. Monitor the bottle with hard-shell and paper-shell cutie pies while bottle is in place on the stainless steel cap.
- b. Slip a clean polyethylene bag around the bottle, and unscrew the bottle carefully.
- c. Screw a polyethylene cap on the bottle, and place the bottle in a bird cage.

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