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UNIT OPERATIONS SECTION MONTHLY PROGRESS REPORT

FEBRUARY 1962

UNIT OPERATIONS SECTION MONTHLY PROGRESS REPORT

February 1962

CHEMICAL TECHNOLOGY DIVISION

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ABSTRACT

In the operation of 6-in.-diameter foam-liquid columns, increase
in either the liquid flow or foam flow caused increased channeling
with increased HTU. Flooding values for the Immi mixer-settler were
obtained using the amine extraction final cycle plutonium flowsheet.
Two tests of the addition of uranyl nitrate to a thoria sol were made,
one of which gave a good product and the other a product which disinte-
grated during calcination. Material balances for 22 waste calcination
runs are summarized.

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SUMMARY

1.0 CHEMICAL ENGINEERING RESEARCH

The heights of transfer units in a 6-in.-ID foam-liquid column were 0.8-1.6 in. for eight countercurrent run conditions including counter-current lengths of 4 to 11 in. and liquid flow rates of 40-100 gal/hr ft^2. As the liquid flow is decreased or the foam flow is increased, channeling decreases and therefore the HTU_x values decrease. Increases in HTU_x values occur when the distribution of liquid feed results in a localized channeling.

2.0 SOLVENT EXTRACTION STUDIES

Flooding values for the Immi mixer-settler were obtained using the amine extraction final cycle plutonium flowsheet. Flooding due to insufficient pumping occurred in the extraction-scrub bank at flows greater than 3.85 gal ft^{-2} min-1 and 1600 rpm impeller speeds. Flooding in the stripping bank occurred at flow rates above 1.71 gal ft^{-2} min^{-1} and 1600 rpm impeller speeds. Flooding in the solvent treatment bank occurred at flow rates greater than 1.85 gal ft^{-2} min^{-1} and 1400 rpm impeller speed. Batch phase separation studies showed that the dispersions similar to those formed in either pulse columns or mixer-settlers under these flowsheet conditions had satisfactory primary break times.

3.0 THORIUM UTILIZATION STUDIES

Two tests of the addition of uranyl nitrate to a thoria sol gave one good ThO_2-UO_2 product and one that disintegrated during calcination. Crushing and sizing of the dried gel is an effective way to obtain a 6-16 mesh size fraction calcined product, but the 70-100 and < 200 mesh material formed sintered agglomerates during calcination. The kilorod experimental test facility furnace operated satisfactorily, but alterations appear necessary to improve sealing during reduction and to decrease the cool down time.

4.0 WASTE PROCESSING

Automatic control of the continuous evaporator-calciner complex maintained good control for over 90% of the time for a double fill test (R-64), which required 49 hrs of total operating time.

Material balances for the past 22 tests are summarized, with 88% balance for major components overall. Complete rate data for the 22 tests are presented with bulk densities of calcined cake and off-gas volumes.

1.0 CHEMICAL ENGINEERING RESEARCH

1.1 Foam Separation - P. A. Haas, D. A. McWhirter

Continuous countercurrent and batch operations of a 6-in.-ID foam-liquid column was continued to study engineering variables controlling column performance. Experimental runs were made to determine the height of a transfer unit as a function of column length, flow rates, and foam bubble sizes.

Countercurrent Run Results. Results are tabulated for three runs (Table 1.1) to investigate the effects of column length, and flow rates on the heights of transfer units. The feed Sr concentrations were all 2×10^{-6} $\underline{M}$, the surfactant-complexing agent was dodecylbenzenesulfonate, and the foam was generated by N_2 through the 50μ dia holes of a spinnerette. The tracer was Sr-89 containing 4-9% Sr-90 and its daughter Y-90. The effect of Y-Sr separation was eliminated by waiting two weeks to determine gross β concentrations or the effects were checked by check analyses made 3 or 4 weeks after a run was completed. The condensed foam was acidified and diluted by a 0.1 $\underline{N}$ HCl purge to the foam breaker and the actual condensed foam volume rates are not accurate to better than $\pm$ 2 cc/min.

The strontium distribution coefficients were calculated from the exit gross β activities assuming one theoretical stage for the runs with zero countercurrent length. This value of Γ/c was then used for the remaining parts of the same run. The values of x_2^* and x_1 were calculated from equilibrium and material balance relationships. The equations where $a = 6/d$ are:

$$\left(\frac{\Gamma}{c}\right)\left(\frac{V}{L}\right) = \left(\frac{\Gamma}{c}\right)\left(\frac{V}{a}\right)\left(\frac{6}{d}\right)\left(\frac{1}{L}\right) = \frac{y_2 V}{x_2^* L}$$

For one theoretical stage (zero countercurrent length):

$$\left(\frac{\Gamma}{c}\right)\left(\frac{V}{L}\right) = \frac{\text{Sr out in foam (surface), cpm/min}}{\text{Sr out in liquid, cpm/min}} = \frac{y_2 V}{x_B L}$$

From a liquid pot material balance with $L_B = 0$:

$$x_1 = \left[1 + \left(\frac{\Gamma}{c}\right)\left(\frac{V}{L}\right)\right] x_B - \left(x_1 - x_B\right) \frac{E_1}{L} \approx \left[1 + \left(\frac{\Gamma}{c}\right)\left(\frac{V}{L}\right)\right] x_B$$

Then the number of transfer units based on the liquid phase were calculated from:

$$N_x = \frac{(x_2 - x_1)}{(x - x^*)_{\text{ln mean}}}$$

The results show HTU_x values of 0.8 to 1.6 in., but the effects of the several variables are combined. From the results and from observation

Table 1.1. Foam Column Material Balances and HTU Calculations - Runs 15, 16, and 17

Surfactant Complexing Agent: Dodecylbenzenesulfonate, concentrations given are ppm of Trepolate F-95, about 95% sodium salt.

Gas Sparger: Au-Pt spinnerette with 1794 holes of 50μ dia.

Constant Feed Concentrations: 2×10^{-6} $\underline{M}$ $Sr(OH)_2$; 10^{-3} $\underline{M}$ NaOH in demineralized water from liquid feed.

Liquid Feed Distributor: N_2 gas scrubged with 1 or 0.2 $\underline{M}$ NaOH at 5 psig; $y_B = 0$ cpm/sq cm, $x_B^* = 0$ cpm/cc
"Spiders" of six capillary tubes on 4-in.-dia circle, one tube at center.
Capillaries of 0.061 in. ID for runs 15 and 16, 0.095 in. ID for run 17.

| Quanity | Symbol | Units | Run Numbers | | | | | | | | | | |
			15A	15B	15C	16A	16B	16C	17A	17B	17C	17D	17E
Liquid rate in	$L + E_p$	cc/min	500	500	500	500	500	500	750	1250	500	500	1000
Gas rate	V/a	cc/min	600	600	600	800	800	800	1200	2000	800	800	1600
Condensed foam rate	E_p	cc/min	~1	~1	~1	~1	~2	~2	5	20	~2	~2	12
Net liquid rate	L	cc/min	~500	~500	~500	~500	~500	~500	745	1230	~500	~500	~990
Liquid surfactant conc. in	-	ppm	275	275	275	450	450	450	275	275	275	275	275
Liquid surfactant conc. out	-	ppm	265	258	261	417	400	395	264	256	260	262	259
Condensed foam surfactant rate	-	mg/min	6.1	6.1	8.9	~8	~8	9.5	9.5	16.6	7.3	6.7	14.9
Surfactant material balance	-	%	101	98	101	~96	~92	92	101	98	100	100	100
Countercurrent length	z	cm	0	10	20	0	14	28	27	27	27	0	27
Average bubble dia.	d	mm	0.57	0.57	0.57	0.54	0.54	0.54	0.58	0.6	0.56	0.56	0.57
Gross β tracer activities													
Liquid feed	x_2	cpm/cc	5700	5700	5700	4700	4700	4700	5000	5000	5000	5000	5000
Liquid concentration out	x_B(also x_1^*)	cpm/cc	3160	2010	1900	2700	630	440	204	225	104	2540	154
Surface activity out	$y_2 V$	10^3 cpm/min	1110	1815	1960	1230	1490	1950	3300	5300	2230	1360	4450
Gross β material balance	-	%	94	99	102	110	77	92	91	108	91	105	93
Sr distribution coefficient	Γ/c	10^{-3} cm	5.6	5.6	5.6	5.1	5.1	5.1	6.25	6.25	6.25	6.25	6.25
Phase flow ratio	$(\Gamma/c)(V/L)$	dimensionless	0.70	0.70	0.70	0.91	0.91	0.91	1.04	1.02	1.07	1.07	1.06
Liquid conc. in equil. with y_2	x_2^*	cpm/cc	3160	5140	5580	2700	3280	4290	4230	4300	4160	2540	4180
Liquid entering liquid pot	x_1	cpm/cc	5370	3420	3230	5150	1200	840	416	455	216	5260	317
Liquid concentration charge	$x_2 - x_1$	cpm/cc	330	2280	2470	-450	3500	3860	4580	4545	4780	-260	4680
Average driving force	$(x-x^*)_{ln\ mean}$	cpm/cc	2360	920	505	2225	930	405	433	421	360	2590	406
Number of transfer units	N_x	-	0.1	2.5	4.9	-0.2	3.8	9.5	10.6	10.7	13.2	-0.1	11.5
Height of transfer unit	HTU_x	cm	-	4.0	4.1	-	3.7	2.9	2.5	2.5	2.0	-	2.3

of the column during operation, it appears that a length of column immediately below the feed nozzle is relatively inefficient for contacting the foam and liquid. This length decreases as the foam velocity increases and increases as the liquid inlet velocity increases. This effect would be relatively of less importance as the countercurrent length is increased. Channeling becomes much more noticeable as the liquid flow rate is increased and slightly less noticeable as the foam rate is increased. The increase in HTU_x from 0.8 in. at 500 cc/min of liquid to 1.0 in. at 1250 cc/min liquid for the parts of run 17 show the effects of increased channeling. For the same flow rates and concentrations as run 17, the values of 1.6 in. in run 15 show the effects of higher inlet liquid velocities through the smaller feed capillaries, the effects of lower foam velocities, and some effect from the shorter countercurrent length. The HTU_x value of 1.1 in. for run 16C shows the effect of increased countercurrent length as compared to 1.5 in. for 16B and shows the effect of higher inlet liquid velocity through the smaller capillaries as compared to 0.8 in. for run 17C. The effect of the higher surfactant concentrations for run 16 as compared to runs 15 and 17 are not believed to be appreciable. The results of run 17B show HTU_x values of 1.0 in. for liquid feed rates of ~100 gal/hr ft^2, higher liquid flow rates with approximately the same HTU_x values should be possible for improved liquid distributor designs.

2.0 SOLVENT EXTRACTION STUDIES

A. D. Ryon

Flooding values for the Immi mixer-settler reported this month were obtained using the amine extraction final cycle plutonium flowsheet. Batch phase separation studies of the primary break of various dispersions similar to those produced in the contactor are also reported.

2.1 Immi Mixer-settler Studies - R. S. Lowrie

Work was started this month to evaluate the flow capacity and stage efficiency of the Immi mixer-settlers as a function of operating variables using the amine extraction final cycle plutonium recovery flowsheet. A typical flowsheet for the engineering tests (Figure 2.1) used 1.5 $\underline{M}$ nitric acid containing 0.05 $\underline{M}$ SO$_4$ as the feed, 5.0 $\underline{M}$ nitric acid as the scrub, 0.3 $\underline{M}$ trilauryl amine in diethylbenzene as solvent* and 2.0 $\underline{M}$ acetic acid as the stripping reagent. The solvent was treated with 1 $\underline{M}$ sodium carbonate solution before recycle back to the process. Properties of the solutions used in the tests are shown in Table 2.1. Lack of containment precluded testing the flowsheet with plutonium.

2.2 Description of Immi Mixer-settler

The Immi mixer-settler assembly available for test purposes consisted of "A" bank, a 17-stage extraction-scrub unit, "B" bank, a 17-stage stripping unit, and "C" bank, an 11-stage solvent treatment unit, racked vertically for gravity flow of the solvent from "A" to "B" to "C". The banks were identical in size; each mixing chamber was 1-in. by 1-in. by 2-1/2-in. deep and each settling chamber was 1-in. by 4-in. by 3-1/8-in. deep (Figure 2.2). Aqueous flow through the contactor was governed by the pumping action of the impellers which pulled aqueous into the bottom of the mixing chamber from the adjacent upstream settler through a hooded port. The solvent stream flowed into the top of the mixer by gravity from the adjacent downstream settler. The dispersion formed was discharged through louvered ports to the settler chamber. The raffinate stream was discharged through the gravity leg controlling the interface in the last settler.

2.3 Flooding Rates for Extraction and Stripping Contactors

Immi mixer-settler units can flood either by dispersion carryover through the solvent overflow port or by insufficient pumping of the aqueous phase in the mixer. Since both types of flooding are dependent on the impeller speeds, there will be, for any given flow rate, a minimum impeller speed (reported as rpm) to prevent flooding due to insufficient pumping and a maximum speed above which flooding due to dispersion carryover occurs. Flow capacity studies (Table 2.2) carried out in the "A" bank using 8 scrub and 9 extraction stages showed that flooding due to insufficient pumping occurred at 1.28 gal ft^{-2} min^{-1} (total flow of both phases in gal per min

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* The diluent and amine used to make up the solvent are described in the October Unit Operations Monthly Report, ORNL-TM-121.

Feed - 1.5 M HNO$_3$
(100) 0.05 M SO$_4$

Solvent
(25)
0.3 M Alamine 304
in Diethylbenzene

Scrub
(10)
5.0 M
HNO$_3$

Extraction - Scrub Contactor

9 Extraction   8 Scrub
Stages   Stages

Raffinate

Loaded
Solvent
(25)

Strip
Reagent
(5.0)
2.0 M Acetic Acid

Stripping Contactor

17 Stages

Plutonium
Product

Stripped
Solvent

Solvent Treatment Contactor

11 Stages

1 M Sodium
Carbonate
7.5

Spent
Carbonate

Treated
Solvent
(Recycled)
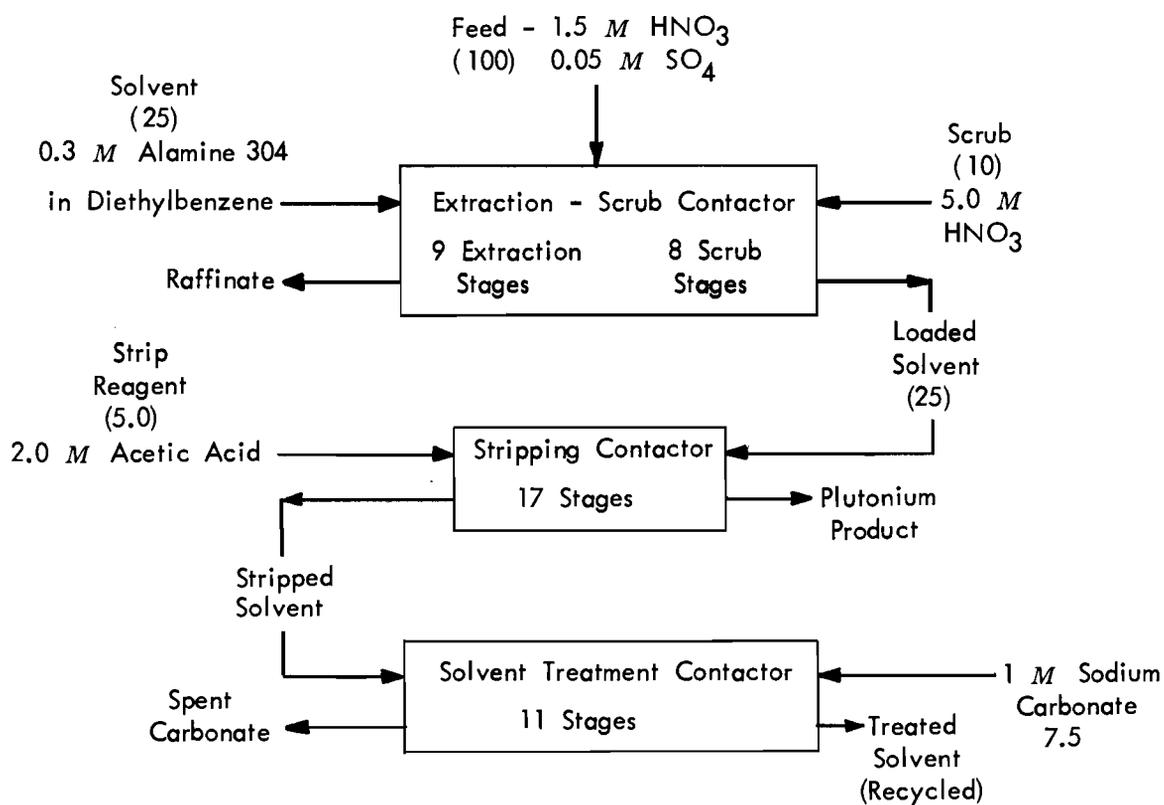
Fig. 2.1. Amine extraction flowsheet for final cycle plutonium recovery.

Table 2.1. Properties of Test Solutions

Analysis	Feed	Scrub	Strip	Carbonate	Solvent
HNO_3, $\underline{M}$	1.5	5.0			
SO_4, $\underline{M}$	0.05				
Acetic Acid, $\underline{M}$			2.0		
CO_3, $\underline{M}$				1.0	
Density, g/cc	1.05	1.17	1.01	1.09	0.85
Viscosity, cps	0.90	1.12	1.05	1.37	1.29
Amine, $\underline{M}$					0.32
Flash point open cup, °C					64°

Density and viscosity measured at 25°C.

Table 2.2. Flooding Tests in Immi Mixer-Settlers

Total Flow gal ft^{-2} min^{-1}	Minimum Stirrer Speed to Prevent Flooding due to Insufficient Pumping, rpm	Maximum Stirrer Speed Before Flooding Due to Dispersion Carryover, rpm
Extraction Bank - 8 stages scrub - 9 stages extraction		
1.28	900	> 2300
2.57	1000	> 2300
3.21	1200	> 2300
3.85	1600	> 2300
4.49	> 2300	-
Stripping Bank - 17 stages		
1.14	900	1600
1.71	1200	1600
2.28[a]	> 1600	1600
Solvent Treatment Bank		
1.24	1200	> 2000
1.85	1400	> 2000
2.47	> 2000	> 2000

[a] Flooding due to both insufficient pumping and dispersion carryover.

ORGANIC IN

ORGANIC OUT

AQUEOUS OUT

SETTLING CHAMBER

MIXED PHASE
TRANSFER PORT

AQUEOUS IN

MIXING CHAMBER

Fig. 2.2. Typical Immi mixer settler stage.

per sq ft of settler area) at impeller speeds below 900 rpm. Increasing the flow rate also increased the minimum impeller speed until at 4.49 gal ft^{-2} min^{-1}, the required impeller speed exceeded the 2300 ppm tested. No flooding due to dispersion carryover was noted at any flow rate with impeller speeds up to 2300 rpm during extraction.

The "pinch" between flooding due to insufficient pumping and flooding due to dispersion carryover severely limited the flow of the 17-stage Immi used as a stripping contactor. Impeller speeds above 1600 rpm caused flooding due to dispersion carryover at all flow rates tested. The minimum speed necessary to prevent flooding due to insufficient pumping was 900 rpm at 1.41 gal ft^{-2} min^{-1}, increasing to 1200 rpm at 1.71 gal ft^{-2} min^{-1}. At 2.28 gal ft^{-2} min^{-1} flow rate and 1600 rpm impeller speed, the unit flooded due to both dispersion carryover and insufficient pumping.

The mixers in both banks tended to operate with the major phase continuous, regardless of which phase was continuous at startup.

2.4 Flooding Rates for the Solvent Treatment Contactor

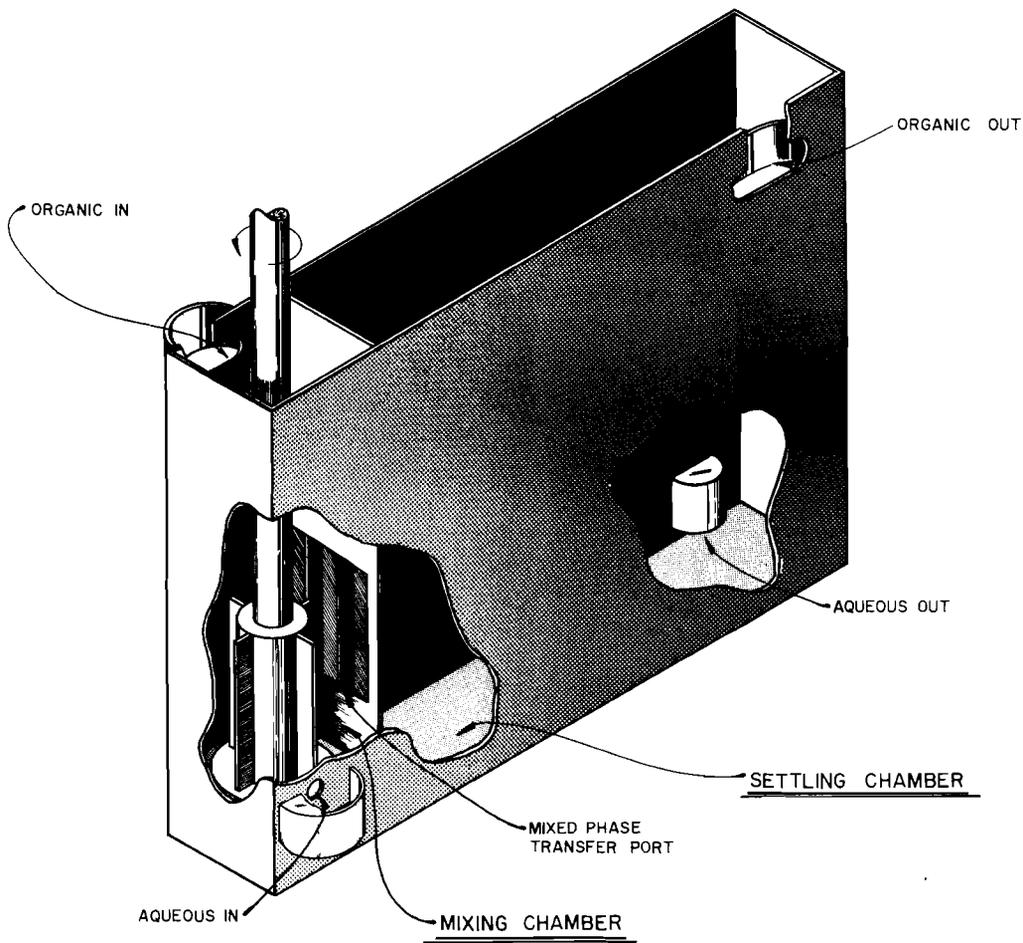In general, an alkaline wash treatment is required to clean up the stripped solvent before recycle, although neither the degree of solvent cleanup nor the reagent have been specified for this flowsheet. Solvent coming from the stripping step contains the equivalent of 0.6 $\underline{N}$ acid (as both free acid and the amine salt) which is neutralized by the sodium carbonate. Flooding tests were carried out in a 17-stage Immi. The stripped solvent was introduced into the 11th stage mixer and the 1 $\underline{M}$ sodium carbonate into the 1st stage mixer. Interface control in the 11th stage settler was provided by adjusting the gravity leg on the 17th-stage settler and was consequently quite poor. Flooding due to insufficient pumping occurred at impeller speeds below 1200 rpm at a flow rate of 1.24 gal ft^{-2} min^{-1}. Increasing the flow rate to 1.85 gal ft^{-2} min^{-1} increased the minimum speed to 1200 rpm. A further increase in flow rate to 2.47 gal ft^{-2} min^{-1} required speeds in excess of 2000 rpm, the highest speed tested. No flooding due to dispersion carryover occurred at impeller speeds up to 2000 rpm. The majority of the CO_2 was released in the second stage from the bottom and did not appear to affect the operation of either the mixer or settler in any way.

2.5 Batch Phase Separation Studies

Very little information was available concerning the physical properties of the solutions or the rate of primary break of the dispersions formed in the amine extraction final cycle plutonium process. Accordingly, batch phase separation tests were made with solvent-aqueous pairs simulating the phase ratio, compositions, etc., found in the end stages of the contactors. Three-hundred ml (total volume) of solution were mixed in a 3-in.-dia baffled beaker for 3 min and the primary break time as well as physical properties were measured (Table 2.3).

Break times for solution pairs simulating the ends of the extraction contactor showed satisfactory break times, based on the assumption that

Table 2.3. Batch Phase Separation Test Data

Solution Pairs	Total H$^+$ M	Acetate M	Nitrate M	CO$_3$ M	Density g/cc	Viscosity cps	Interfacial Tension dynes/cm	Primary Break Time, sec			
								Aq. Cont. Dispersions		Solv. Cont. Dispersions	
								4/1 (A/O)	1/1 (A/O)	1/1 (A/O)	1/4 (A/O)
Fresh Solvent	0.63	-	0.63	-	0.87	2.41	20.4				
Raffinate	1.16	-	1.16	-	1.05	0.89		55	80	130	140
Loaded Solvent	0.78	-	0.78	-	0.87	2.33	22.8				
5.0 M HNO$_3$ Scrub	4.19	-	4.19	-	1.13	1.07		40	65	65	65
Loaded Solvent	0.49	0.19	0.29	-	0.87	2.58	16.7				
Loaded Strip	1.84	1.68	0.09	-	1.01	1.02		120	120	90	120
Stripped Solvent	0.52	0.23	0.29	-	0.87	2.60	15.5				
Stripped Reagent	1.84	1.84	< 0.005	-	1.01	1.03		> 300	> 300	100	100
Stripped Solvent	0.52	0.20	0.30	-	0.87	2.60					
Strip Reagent (2.0 M acetic + 0.1 M HNO$_3$)	1.98	1.84	0.06	-	1.01	1.03	15.1	-	190	90	-
Stripped Solvent	0.004	-	-	-	0.85	1.35	16.4				
Spent Carbonate	-	0.52	0.82	0.24	1.08	1.10		30	45	35	35
Treated Solvent	-	-	< 0.005	-	0.85	1.28	< 1.0				
1 M Sodium Carbonate	-	-	< 0.005	1.0	1.09	1.40		50	60	170	170

dispersion which required more than 300 sec to break would probably cause flooding at very low throughput. Density, viscosity, and interfacial tension values did not change appreciably.

Primary break times for the stripping contactors were satisfactory for the bottom (product) end but were > 300 sec for aqueous continuous dispersions at the top (stripped solvent). However, the addition of 0.1 M nitric acid to the 2.0 M acetic acid strip reagent reduced these break times to < 200 sec. Further, the aqueous continuous dispersion was quite sensitive to the amount of mixing and merely decreasing the mixing energy (lower stirrer speed) produced dispersions breaking in < 300 sec. Viscosity, density, and interfacial tension values were virtually unchanged in passing through the strip contactor.

Both solvent continuous and aqueous continuous dispersion typical of those formed in the solvent treatment contactor broke quite rapidly although the break times, particularly the solvent continuous dispersions, increased as the amine changed from the acid salt to the free amine. This change also explains both the decrease in viscosity of the solvent from 2.6 to 1.3 cps and the drop in interfacial tension from 16.4 to < 1.0 dynes/cm. Several tests were made to obtain some idea of how much Na_2CO_3 could be neutralized before the surface and interfacial tensions started to increase. Small amounts of nitrate, also chloride and sulfate had no affect on surface tensions of the aqueous solution before equilibrations with solvent (Table 2.4) and, in the nitrate case, no affect on surface tensions or interfacial tension after equilibration with solvent (Table 2.5). Neutralization of ~25% of the carbonate with nitrate did not affect the surface tension of the aqueous solution before solvent contact, but did increase both surface tension (from 25.6 to 42.6 dynes/cm) and interfacial tension (from < 1.0 to 10.4 dynes/cm) after equilibration with solvent.

Table 2.4. The Affect of Small Amounts of

Nitrate, Chloride, and Sulfate on

Surface Tensions of 1 M $NaCO_3$ Solutions

Acid Present	M	Surface Tension at 25°C dynes/cm
None	–	71.2
HNO_3	0.07	71.2
HCl	0.05	70.8
H_2SO_4	0.08	71.2

Table 2.5. The Effect of Nitrate on Surface and Interfacial Tension Values

of Solvent and 1 $\underline{M}$ NaCO$_3$ Solutions

Test Solutions	NO$_3$ Conc., $\underline{M}$	Break Time Sec (A/O = 1)		Surface Tension, dynes/cm		Interfacial Tension dynes/cm
		Solvent Continuous	Aqueous Continuous	Before Equilibration	After Equilibration	
Aqueous Solvent	0 0	140	68	71.6 27.2	25.6 27.6	< 1.0
Aqueous Solvent	0.05 0	137	77	71.6 27.2	25.6 26.5	< 1.0
Aqueous Solvent	0.53 0	107	72	71.0 27.2	42.6 27.0	10.4

A method for cooling the furnace in the required time is also necessary because the effect of quick cooling on the furnace refractories should be determined. A metal plate pressed securely against the front of the furnace muffle (with the regular furnace [insulating] door in a raised position) allows cooling in 12 hr. Accordingly, a false door, or air lock, is being designed which will completely enclose the front of the furnace. The present insulated furnace door will be enclosed in all positions by this air lock. During high temperature operation the insulated door will be in the closed position. During cool down, the door will be raised stepwise thus gradually exposing the furnace muffle to the inside of the air lock. The rate at which the furnace cools down in the higher temperature ranges (where refractory damage will most likely occur) will be controlled by positioning the insulated door. The new air lock will also provide a gas-tight seal for the door; the present sealing arrangement is inadequate. The rear of the furnace will be sealed by covering with a metal plate.

The furnace cycle time could also be reduced by removing the product from the furnace at 400°C instead of the presently accepted 200°C. A box could be designed which would contain an inert atmosphere and into which the product could be drawn. An estimated 5 hr might thus be saved. As this is a fixture which can be designed readily and with reasonable assurance of operability, it will not be built for mock-up operation unless a need is demonstrated.

During the revisions on the furnace, it was found difficult to turn the furnace around and disconnect the services. A minimum clearance of 4 in. over the diagonal dimension is felt necessary. Services at the rear of the furnace should be brought out through the side of the furnace facing the nearest point of accessibility and provided with quick disconnects. The furnace must also be securely bolted in place to facilitate removal of heavy crucibles.

The vibratory compaction densities obtained on the products of the facility appeared satisfactory (Table 3.1). Of the batches listed which were crushed by an arbitrary procedure following firing, i.e., batch Nos. 1 (A and B), 2 (A and B), and TDNC, only the TDNC batch yielded the required minimum of 60 wt % of +16 mesh material. Until a larger number of firings have been made, however, the severity of this problem cannot be assessed.

A time-temperature sequence circuit which automatically executes the entire furnace program was installed. This device permits almost continuous use of the equipment and has operated faultlessly thus far.

Alumina crucibles (6-in.-OD x 5-in.-ID x 4-in.-deep) have given excellent service. It would be desirable to have crucibles with somewhat flattened sides for more efficient use of furnace space.

Table 3.1. Properties of Sol-Gel Materials

Prepared in the Test Facility

Batch No.	Wt % U	O/U Ratio	Sorbed Gases (cc/g-1200°C)	Vibratory Comp. Density, g/cc	
				5/16-in.-dia Tube*	1/2-in.-dia Tube
1A	2.99	2.07	0.053	8.30	8.87**
1B	3.04	2.12	0.049		
2A	3.19	2.06	0.034	8.48	8.83**
2B		2.16	0.083		
3				-	8.88
TDNC	3.02	2.15	-	8.35	-
42-43	3.22	2.40	-	8.45	8.59***
					8.56***

   * Vibrated on 1-1/4-in. Navco vibrator.
  ** Vibrated on 1-1/2-in. Branford vibrator.
 *** Vibrated on 1-5/8-in. Navco vibrator.

4.0 WASTE PROCESSING

J. C. Suddath

The purpose of the waste processing program is to provide engineering data and experience for the design of the waste pot calciner pilot plant. A number of tests, R-42 to R-64, were completed in the last half of 1961. The data for these tests have been compiled and will be reported for the next few months as it is processed.

4.1 Material Balances for Tests R-42 to R-64 - C. W. Hancher

Detailed material balances for tests R-42 through R-64 have been completed (Tables 4.1 and 4.2). The following basis was used for the calculation:

Feed: The concentrations were averaged and multiplied by the system feed volume.

Evaporation: The starting and ending evaporator concentration and volume were multiplied respectively and then the difference was taken.

Condensate: Since the system did not have an installed accurate method of determining the condensate volume flow, it was assumed equal to feed plus the purge and evaporator water. Forty liters were subtracted from the first 2 hrs to compensate for the initial feeding of the pot and evaporator.

Solid: The analytical results for three solid samples, top, middle, and bottom, were averaged and multiplied by the total weight.

4.2 Material Balance Results, Tests R-42 to R-64

The average of all major ion material balances was 88% (Table 4.3). All of the ion material balances except Hg were 91% or better. Mercury should be low because large quantities of yellow mercury salts were deposited on the off-gas piping.

In the TBP-25 tests where iron was a minor constituent, the iron balances as a rule were much above 100% because of poor sampling of the solid and/or poor analytical accuracy.

4.3 Control of Test R-64

Test R-64 was a TBP-25 test with an average feed rate of 11.6 liters per hour. Total feed used was 560 liters. The material balance for nitrate and aluminum was 77% and 89%. This was a two-part test: feed for 19 hr, 400 liters; calcine for 8 hr; feed for 18 hr more, 160 liters; and finally calcine for 13 hr (Table 4.4). The resulting solid had a bulk density of 0.80 g/cc.

Table 4.1. Material Balances for Waste Calciner Tests R-42 through R-64

	HNO$_3$ g	HNO$_3$ %	Al g	Al %	Fe g	Fe %	Hg g	Hg %	SO$_4$ g	SO$_4$ %	Ru g	Ru %	P g	P %	Ca g	Ca %
								Purex Run R-42								
IN																
Feed	195,722				14,270				48,311		79.28				12,397	
Added	41,131															
Total	236,853															
OUT																
Evap.	3,500	1.48			655	4.6			2,157	4.4	5.9	7.44				
Cond.	248,372	104.8			89.3	0.6			483	1.0						
Solid	685.8	0.29			12,807	89.7			44,280	91.49	38.7	48.8			13,500	108.9
Total		106.5				94.9				96.9		56.24				108.9
								Purex Run R-43								
IN																
Feed	218,592				15,503				54,826		74.84				12,608	
Added	3,392															
Total	221,984															
OUT																
Evap.	117	0.1			51.6	0.33					2.55	3.41				
Cond.	181,474	81.7			128.4	0.83			548	1.0						
Solid	210	0.1			9,949	64.0			43,548	80.3	31.36	42				
Total		81.9				65.2				81.3		45.41				

Table 4.1. Continued

	NO₃		Al		Fe		Hg		SO₄		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%
Purex Run R-44																
IN																
Feed	146,422	100			10,838	100					50.31	100			13,629	100
Added																
Total	146,422															
OUT																
Evap.	1,482	1.01			1,360	12.5					7.73	15.35			650	4.7
Cond.	175,698	120			42.8	0.39					8.72	17.33			80	0.6
Solid	64.2	0.04			8,983	83.0					22.77	45.3			13,800	101.3
Total		121.05				95.89						78				106.6
Run R-45																
IN																
Feed	114,472				11,283										9,450	
Added	28,272															
Total	142,744															
OUT																
Evap.	-25,630	-18.0			1,825	16.2										
Cond.	172,125	121.0			22.4	0.2										
Solid	3,959	2.77			6,433	57.0									9,252	97.9
Total		105.7				73.4										97.9

Table 4.1. Continued

	NO$_3$		Al		Fe		Hg		SO$_4$		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%
Purex-Batch Run R-46																
IN																
Feed	133,575				11,180										11,400	
Added	51,875	100														
Total	185,450															
OUT																
Evap.	-25,176	-13.57			498	4.5										
Cond.	179,631	97.0			5.6	0.05									40	0.35
Solid	948.8	0.512			11,386	102									20,874	183
Total		83.9				106.5										183.35
TBP-25 Run 47																
IN																
Feed	176,748	100	20,763		68.21		1,857									
Added																
Total	176,748															
OUT																
Evap.	10,550	5.9	211.3	0.17	6.7	9.8	192	10								
Cond.	135,503	77.0	1,242	5.9	13.2	19.3	197	10.6								
Solid	134.33	0.076	17,947	86.0	212.6	312	356	19								
Total		82.9		92.1		341.2		40.0								

Table 4.1. Continued

	HNO₃		Al		Fe		Hg		SO₄		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%
TBP-25 Run R-48																
IN																
Feed	203,112	100	22,089	100	76.75	100	2,049	100								
Added																
Total	203,112	100														
OUT																
Evap.	12,900	6.35	1,015	4.6	7.13	9.2	270	13								
Cond.	203,112	100					164	8								
Solid	46.72	0.023	18,711	84.7	292.0	380	756	37								
Total		106.37		89.3		389.3		58								
TBP-25 Run R-49																
IN																
Feed	209,364	100	24,712		124.3		2,772									
Added																
Total	209,364															
OUT																
Evap.	10,350	4.0	1,370	5	8.6	7.0	162	6								
Cond.	149,274	71.3	26.9	0.1	7.8	6.3	56	2								
Solid	1,740	0.83	28,750	116	150	121	310	11								
Total		76.13		121.1		134.1		19								

Table 4.1. Continued

	HNO$_3$ g	HNO$_3$ %	Al g	Al %	Fe g	Fe %	Hg g	Hg %	SO$_4$ g	SO$_4$ %	Ru g	Ru %	P g	P %	Ca g	Ca %
TBP-25 Organic Run R-50																
IN																
Feed	158,122	100	17,576		69.2		1,591				83.04		114			
Added																
Total	158,122															
OUT																
Evap.	12,075	7.64	1,415	8.1	13.2	19.2	206	13			6.675	8.04				
Cond.	123,052	77.8	14.2	0.08	2.84	4.1	60	3.7			1.58	1.9	1.4	1		
Solid	647.9	0.41	13,020	74.1	77.5	120	2,154	134			41.85	50.4	62	54.3		
Total		85.85		82.3		143.3		150.7				60.34		55.3		
TBP-25 Batch Organic Run R-51																
IN																
Feed	122,584	100	14,137		60.9		1,232				64.68		77			
Added																
Total	122,584															
OUT																
Evap.	44,400	36.22	5,655	40	104.1	170.6	1,889.6	153.3			55.8	86.3				
Cond.	120,169	98.03	17.2	0.12	2.1	0.03	83.65	6.79			2.87	4.44	0.08	1		
Solid	1,062	0.866	15,222	107.7	106.2	174.2	531	43.1			60.18	93	77	100		
Total		135.12		147.4		344.8		203.1				183.74		101		

Table 4.1. Continued

	HNO$_3$		Al		Fe		Hg		SO$_4$		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%

TBP-25 Run R-48

IN																
Feed	203,112	100	22,089	100	76.75	100	2,049	100								
Added																
Total	203,112	100														
OUT																
Evap.	12,900	6.35	1,015	4.6	7.13	9.2	270	13								
Cond.	203,112	100					164	8								
Solid	46.72	0.023	18,711	84.7	292.0	380	756	37								
Total		106.37		89.3		389.3		58								

TBP-25 Run R-49

IN																
Feed	209,364	100	24,712		124.3		2,772									
Added																
Total	209,364															
OUT																
Evap.	10,350	4.0	1,370	5	8.6	7.0	162	6								
Cond.	149,274	71.3	26.9	0.1	7.8	6.3	56	2								
Solid	1,740	0.83	28,750	116	150	121	310	11								
Total		76.13		121.1		134.1		19								

Table 4.1. Continued

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	HNO$_3$ g	HNO$_3$ %	Al g	Al %	Fe g	Fe %	Hg g	Hg %	SO$_4$ g	SO$_4$ %	Ru g	Ru %	P g	P %	Ca g	Ca %
TBP-25 Organic Run R-50																
IN																
Feed	158,122	100	17,576	69.2			1,591				83.04		114			
Added																
Total	158,122															
OUT																
Evap.	12,075	7.64	1,415	8.1	13.2	19.2	206	13			6.675	8.04				
Cond.	123,052	77.8	14.2	0.08	2.84	4.1	60	3.7			1.58	1.9	1.4	1		
Solid	647.9	0.41	13,020	74.1	77.5	120	2,154	134			41.85	50.4	62	54.3		
Total		85.85		82.3		143.3		150.7				60.34		55.3		
TBP-25 Batch Organic Run R-51																
IN																
Feed	122,584	100	14,137	60.9			1,232				64.68		77			
Added																
Total	122,584															
OUT																
Evap.	44,400	36.22	5,655	40	104.1	170.6	1,889.6	153.3			55.8	86.3				
Cond.	120,169	98.03	17.2	0.12	2.1	0.03	83.65	6.79			2.87	4.44	0.08	1		
Solid	1,062	0.866	15,222	107.7	106.2	174.2	531	43.1			60.18	93	77	100		
Total		135.12		147.4		344.8		203.1				183.74		101		

Table 4.1. Continued

	HNO₃ g	HNO₃ %	Al g	Al %	Fe g	Fe %	Hg g	Hg %	SO₄ g	SO₄ %	Ru g	Ru %	P g	P %	Ca g	Ca %
							TBP-25 Batch Organic Run R-52									
IN																
Feed	213,840	100	23,188		136.4		3,190				114.4		127			
Added																
Total	213,840															
OUT																
Evap.	43,266	20.23	8,923	38.5	126	92.5	951	29.8			37.5	32.78				
Cond.	26,812	17.66														
Solid	23.76	0.01	12,408	53.5	158.4	116.1	211.2	6.6			58.08	50.77	52.8	41.5		
Total		38.0		92.0		208.5		36.4				83.55		41.5		
							TBP-25 Run R-54									
IN																
Feed	168,632	100	16,264		77.04		1,510.8				81.32					
Added																
Total	168,632															
OUT																
Evap.	12,400	7.35	1,187	7.3	42.0	54.2	522.5	34.6			5.05	6.21				
Cond.	181,792	107.8	2.9	0.02	0.1	0.13	41.9	2.77			1.51	1.86				
Solid	167.7	0.099	18,135	112	187.2	243.0	278.85	18.46			68.25	84				
Total		115.25		118.8		297.6		55.83				92.1				

Table 4.1. Continued

	HNO$_3$		Al		Fe		Hg		SO$_4$		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%

TBP-25 Run R-55

	HNO$_3$		Al		Fe		Hg		SO$_4$		Ru		P		Ca	
IN																
Feed	196,980		18,901		178.2		2,875				89.1					
Added																
Total	196,980															
OUT																
Evap.	12,875	6.54	1,395	7.4	26	14.6	442.5	15.4			6.73	7.55				
Cond.	133,842	66.9	72.7	0.38	1.92	1.1	81.87	2.85			1.79	2.01				
Solid	108.3	0.055	17,917	94.8	148.2	83.1	95	3.3			45.6	51.2				
Total		74.5		102.5		98.8		21.55				60.76				

Darex Run R-56

	HNO$_3$		Al		Fe		Hg		SO$_4$		Ru		P		Ca	
IN																
Feed	133,667	100			25,278											
Added																
Total	133,667															
OUT																
Evap.	12,450	9.3			1,212	4.8										
Cond.	87,845	65.7			2.29	0.01										
Solid	25.8	0.02			25,768	97.9										
Total		75.02				102.7										

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Table 4.1. Continued

	HNO$_3$		Al		Fe		Hg		SO$_4$		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%

Darex Run R-57

IN																
Feed	298,706	100			63,971											
Added																
Total	298,706															
OUT																
Evap.	12,550	4.2			1,945	3.04										
Cond.	195,298	65.3				9.68 0.02										
Solid	168.0	0.056			39,480	61.7										
Total		69.60				64.75										

Darex Run R-58

IN																
Feed	216,576	100			40,896											
Added																
Total	216,576															
OUT																
Evap.	14,575	6.73			2,650	6.4										
Cond.	170,168	78.57				3.43 0.01										
Solid	145.18	0.067			40,071	98.0										
Total		85.40				104.5										

Table 4.1. Continued

	HNO₃		Al		Fe		Hg		SO₄		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%
TBP-25 Batch Organic Run R-59																
IN																
Feed	166,740	100	19,572		59.4		1,627.7				79.4					
Added																
Total	166,740															
OUT																
Evap. Cond.	161,579	97	1,320	6.7	6.65	11.2	1,273	78.21			6.65	8.38				
Solid	202.72	0.122	17,792	91			720.4	44.3								
Total		97.122		97.75		11.2		122.51				8.38				
Darex Batch Run R-60																
IN																
Feed	190,176	100			39,313	100										
Added																
Total	190,176															
OUT																
Evap.	42,250	22.2			12,186	30.1										
Cond.	147,633	77.63			2,218	5.64										
Solid	4.64	0.002			31,173	79.3										
Total		99.83				115.04										

Table 4.1. Continued

	HNO₃		Al		Fe		Hg		SO₄		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%

Darex Batch Run R-61

	HNO₃ g	HNO₃ %	Al g	Al %	Fe g	Fe %	Hg g	Hg %	SO₄ g	SO₄ %	Ru g	Ru %	P g	P %	Ca g	Ca %
IN																
Feed	173,762	100			34,691	100					81.355	100				
Added																
Total	173,762															
OUT																
Evap.	104,372	60.1			65.78	0.19					1.628	2.0				
Cond.	85,855	41.41			13.99	0.04					1.53	1.88				
Solid	215	0.12			30,572	88.13					44.35	54.50				
Total		104.63				88.33						58.38				

TBP-25 Run R-62

	HNO₃ g	HNO₃ %	Al g	Al %	Fe g	Fe %	Hg g	Hg %	SO₄ g	SO₄ %	Ru g	Ru %	P g	P %	Ca g	Ca %
IN																
Feed	201,146	100	21,854	100	82.51	100	1,908.8	100								
Added																
Total	201,146															
OUT																
Evap.	13,000	6.46	967.5	4.43	54.75	66.36	440	23.1								
Cond.	129,812	64.53	4.61	0.02			39.14	2.05								
Solid	644	0.32	17,371	79.5	47.3	57.2	1,086	56.9								
Total		71.31		85.95		123.56		82.05								

Table 4.1. Continued

	HNO₃		Al		Fe		Hg		SO₄		Ru		P		Ca	
	g	%	g	%	g	%	g	%	g	%	g	%	g	%	g	%
TBP-25 Run R-63																
IN																
Feed	183,556	100	18,945	100	125.5	100	1,957	100								
Added																
Total	183,556															
OUT																
Evap.	12,025	6.55	920	4.85	23.75	18.9	127	6.5								
Cond.	152,339	83	3.41	0.012			46.9	2.4								
Solid	351.2	0.19	17,796	94.0	232.44	185.3	693.7	35.4								
Total		89.74		98.85		204		44.3								
TBP-25 Run R-64																
IN																
Feed	242,280	100	27,440	100	364	100	2,565	100								
Added																
Total	242,280															
OUT																
Evap.	8,550	3.53	817.5	2.97	9.25	2.54	211.25	8.2								
Cond.	176,908	73	3.41	0.02	41.5	11.40	142.89	5.57								
Solid	30.9	0.013	23,659	86.2	76.3	20.96	276.48	10.7								
Total		76.51		89.19		34.90		24.47								
Cond.	185,397	71	174	0.59	3.3	0.08	2,637	103								

Table 4.2. Other Results for Tests R-42 to R-64

Run No. R-	System feed rate, liters hr	Total feed, liters	Water-feed ratio	Feed time, hr	Calcine time, hr	Bulk density, g/cc	Off-gas, cu ft
42	49.5	495	6.4	10	18	1.5	out
43	48.5	594[a]	91	8	9	1.27	649
44	40.9	409	7.8	10	13	1.14	818
45	25.2	328	3.9	13	12	1.17	730
46	25.8	325	4.1	13	12	1.55	1140
47	30.6	429	3.4	14	10	0.57	825
48	15.0	468	2.8	31	17	0.77	1630
49	17.6	478	2.8	27	10	0.83	905
50	11.5	346	2.4	30	10	0.52	900
51	7.0	308	2.5	44	5	0.59	985
52	9.8	440	2.3	45	10	0.44	1100
54	17.2	428	2.7	25	7	0.65	600
55	19.4	469	3.5	24	4	0.60	655
56	13.6	383	4.1	28	11	0.86	537
57	8.9	641	3.2	72	7	1.4	1410
58	16.5	576	3.0	35	10	1.42	484
59	12.4	397	-	32	10	0.61	506
60	8.6	336	5.5	40	0	1.29	603
61	10.6	307	3.8	29	3	1.13	365
62	16.8	446	4.5	27	15	0.63	432
63	16.3	421	2.3	27	10	0.61	521
64	11.6	560	2.1	38	20[b]	0.80	601

[a] System leaked about 200 liters leaving 394.

[b] Two fill (19, 7, 19, 13).

Table 4.3. <u>Material Balance Summary Results</u>

<u>Runs R-42 to R-64</u>

Nitrate average recovery	92%
Al average recovery	99%
Fe average recovery	160%[a]
Hg average recovery	71%
Average	88%

[a]For Fe as a major ion in Purex and Darex tests   91%

Table 4.4.   <u>Sequence Log Test R-64</u>

Hour	Run hour	Feed	Calcine	Cal Liq Level	Evap. Density	Evap. Liq Level	Evap. Pressure	Evap. Conduct- ivity
11:00	Start	On		Auto	Auto	Auto	Auto	Auto
6:00	19	Off	Start	Auto/man	Auto	Auto/man	Auto	Auto
14:00	27	On	Finish	Auto/man	Auto	Auto/man	Auto	Auto
17:00	30			Man/auto	Auto	Man/auto	Auto	Auto
8:00	45	Off	Start	Man/auto	Auto-man	Auto/man	Auto	Auto
10:00	47			Man/auto	Auto-man	Auto/man	Auto	Auto
13:00	50			Auto/off	Man/off	Man/off	Auto/off	Auto/off
21:00	58		Finish					
Automatic Control Time (hrs)				32	46	38	49	45

The control system used for this test was as follows:

1. Calcine Liquid Level → Cal Feed
2. Evaporator Density → Evaporator Steam
3. Evaporator Liquid Level → Evaporator Feed
4. Evaporator Pressure → Gas Take-off
5. Evaporator Condensate Conductivity → Evaporator Water

(See ORNL-LR-DWG 63205R)

4.4 Calciner Liquid Level Control

A copper filled thermocouple was used to sense the liquid level. The controller was on manual during the two calcining periods of the test. The limits of good control were 20% and 95% of the chart (Table 4.5) with a set point of 60%. The range of the chart is approximately 55 to 65 liters of pot volume. The controller settings were 200% proportional band widths and 240 min reset time. The controller can not operate if the liquid level is not up to the thermocouple probe. During the tests when the controller was operating, the process was under the limits three times for 2.8% of the time and above once for 13.5% of the time (Figure 4.1). A total of 16.3% of the time (32 hrs controlling) the system was not within control limits. The 13.5% above the limit, which means low level in the calciner pot, was the fault of the control valve and not the controller.

4.5 Evaporator Liquid Level Control

The evaporator control had an average deviation from the set point of less than 1% during the 38 hr of automatic control. The proportional band setting was 40% and the reset was 10 min. The evaporator liquid level controlled the addition of feed to the system.

This was out of the desired control zones 9% of the time (Table 4.7), 3% above the limit (3 times) and 6% below (5 times)(Figure 4.2). The evaporator was put on manual control during the first calcination period. During this manual operation, no feed was added to this system, however, the evaporator liquid level did increase to above 90% from calciner condensate causing the conductivity control (water addition to strip nitric acid) to be overridden and cut off. Manual addition of steam and water to the system was used to overcome a mal-operation.

4.6 Evaporator Pressure

The evaporator pressure control was within less than 1% of the set point for 49 hrs of automatic control (Figure 4.3). The control was low only three times for 0.6% of the automatic operating time (Tables 4.4, 4.5, 4.6, and 4.7).

4.7 Evaporator Density Control

The evaporator density was measured in an external pump loop with a bubbler. The control output signal was cascaded to the steam controller

Table 4.5. Test R-64 Control Limits for Acceptable Control

	Set Point	Lower Limit	Upper Limit	Chart Range	
		% of Chart		0% to 100%	
Cal liquid level	60	20	95	55	65 liters
Evap density	30	25	35	1.0	2.0 g/cc
Evap liquid level	50	30	70	7	35 liters
Evap pressure	40	38	42	-5	+5 psig
Evap conductivity	40	25	55	1	3 $\underline{M}$ HNO$_3$

Table 4.6. Control Setting for Test R-64

	Prob Band %	Reset min
Cal liquid level	200	240
Evap density	100	10
Evap liquid level	40	10
Evap pressure	25	1
Evap conductivity	200	10

Table 4.7. R-64 Control Limit Response

	Ave Set Point Deviation %	Set Point	% of Time below Limits, %	Number of Times below Limit	% of Time Above Limit	Number of Times Above Limit	Total Control Time, hr
Cal liquid level	-3.2	60	2.8	3	13.5[a]	1	32
Evap density	< + 1	30	1.5	3	0	0	46
Evap liquid level	< + 1	50	3.6	3	5.4	5	38
Evap pressure	< + 1	40	0	0	0.6	3	49
Evap conductivity	< + 1	40	16	8	18	12	45

[a] Calciner low-feeding system did not deliver enough solution to the calciner.

RUN TIME – hrs

Calciner Liquid – Continuous – TBP

Limits 95–20% Controller Action   Prob.   200%
Reset   240 min

Fig. 4.1.   Waste calcination & evaporation controller – Test 64 – calciner liquid level.

% OF CONTROL SCALE

RUN TIME – hrs

Calciner Liquid – Continuous – TBP

Limits 95-20% Controller Action   Prob.   200%
                                  Reset   240 min

Fig. 4.1.   Waste calcination & evaporation controller – Test 64 – calciner liquid level.   (Cont'd)

RUN TIME – hr
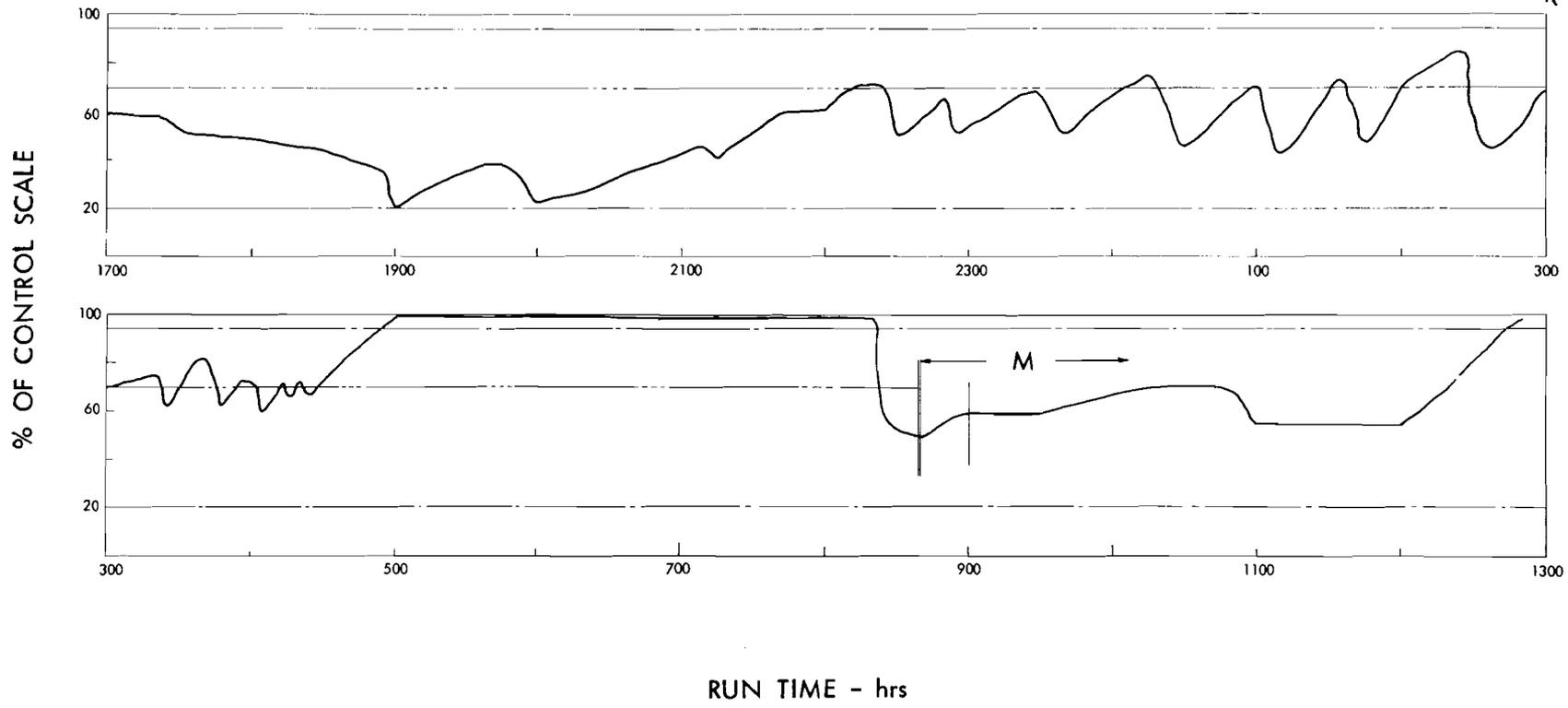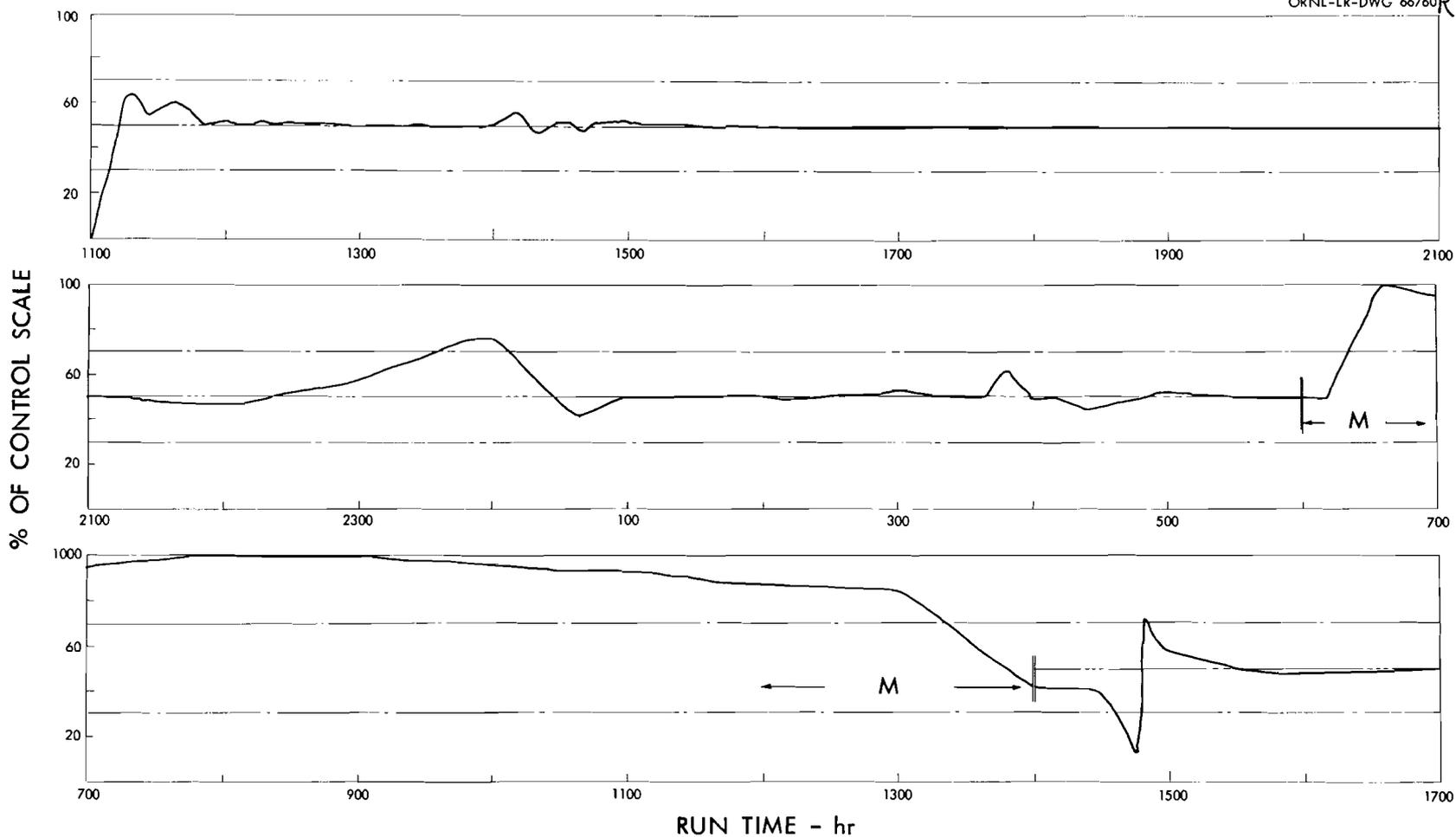
Evaporator Liquid Level – Continuous – TBP-25

Limits SP ± 20% Controller Action   Prob. Band 40%
                                       Reset   10 min

Fig. 4.2. Waste calcination & evaporation control – Test 64 – evaporator liquid level.

Evaporator Liquid Level - Continuous - TBP-25
Limits SP ± 20% Controller Action   Prob. Band 40%
                                      Reset   10 min

Fig. 4.2. Waste calcination & evaporation controller - Test 64 - evaporator liquid level. (Cont'd)
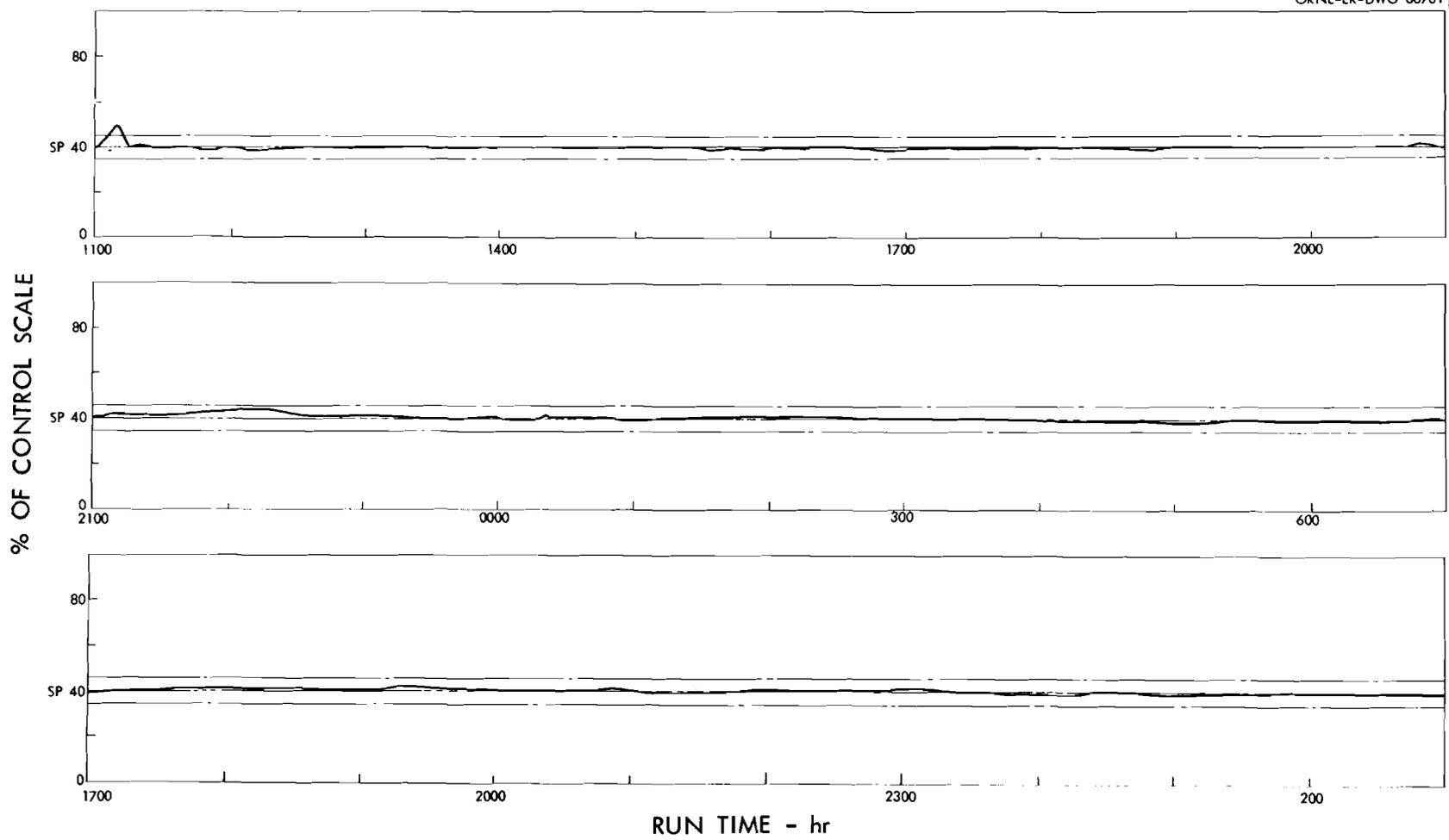
RUN TIME - hr

Evaporator Vapor Pressure - Continuous - TBP-25

Limits SP ±2% Controller Action   Prob. Band 25%
                                    Reset   1 min

Fig. 4.3.   Waste calcination & evaporation control - Test 64 - evaporator vapor pressure.

% OF CONTROL SCALE

RUN TIME – hr
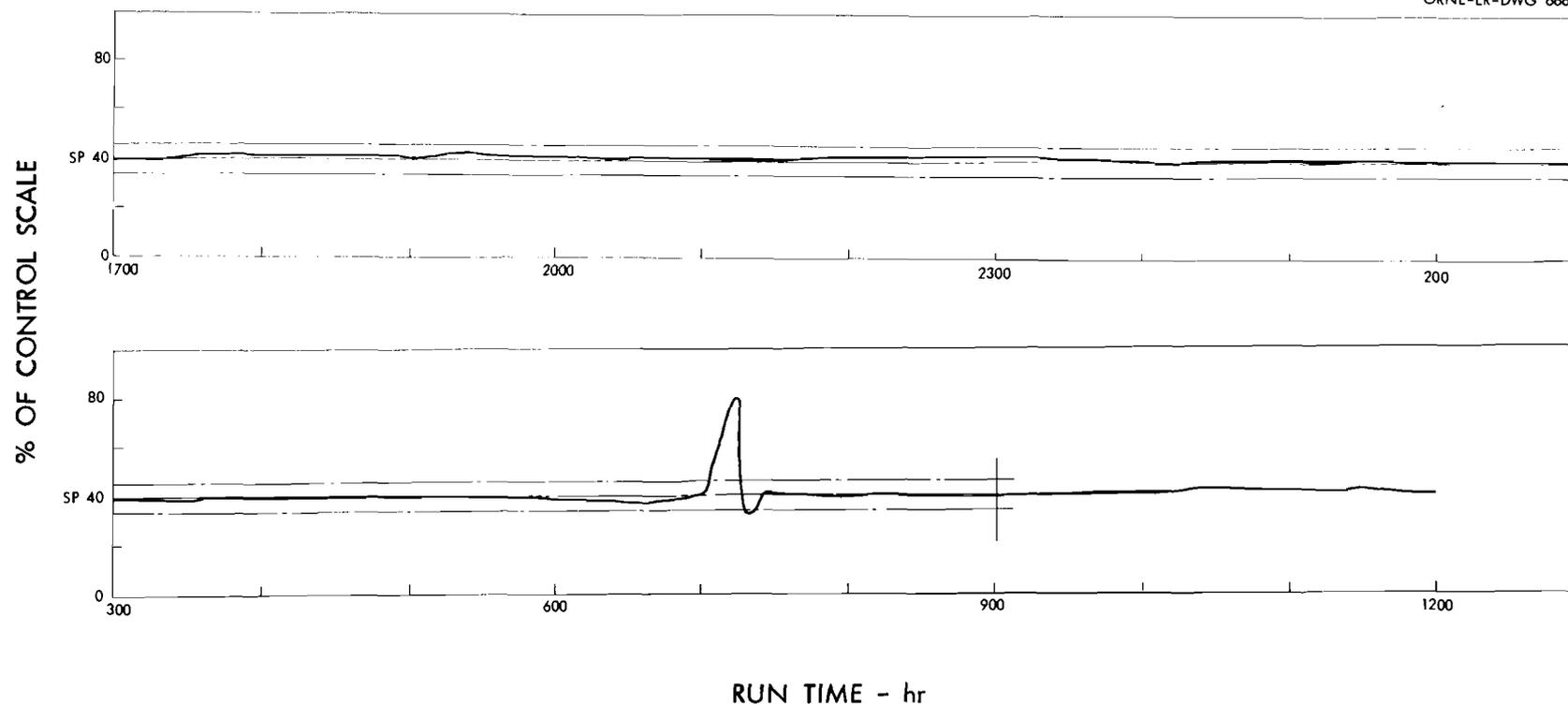
Evaporator Vapor Pressure – Continuous – TBP-25
Limits SP ± 2% Controller Action Prob. Band 25%
Reset   1 min

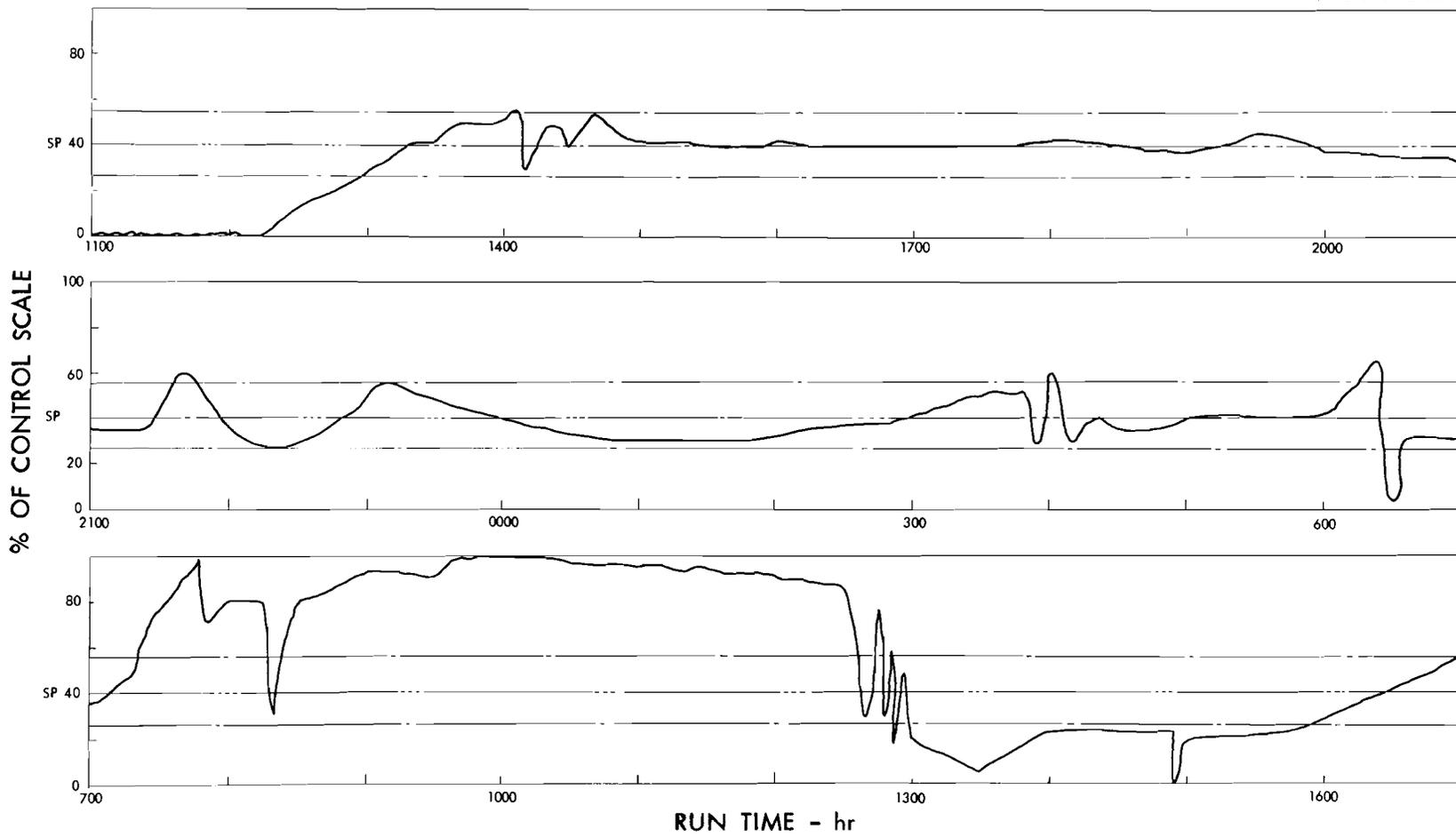Fig. 4.3.   Waste calcination & evaporation control – Test 64 – evaporator vapor pressure.   (Cont'd)

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as a set point. The control was very good. The density was not above
the limits during the test and below the limits three times, 1.5 of 46 hrs
the controller was on automatic (Figure 4.4).

4.8 Evaporator Conductivity

The electrical conductivity of evaporator condensed condensate is
measured to determine the concentration of nitric acid in the evaporator
condensate. The liquid phase nitrate concentration is inferred from the
vapor composition

$$NO_{3_{vap}} = \frac{1}{36} \left(NO_{3_{liq}} \right)^2$$

The nitrate in the liquid should not exceed about $8 \underline{M}$ to maintain minimum
Ru volatility.

The average deviation from the set point was less than 1%. Total %
of time outside of the desired control limits was 34% (16% below, 8 times,
and 18% above, 12 times)(Figure 4.5). Ten of the 16% above the desired
control limits was due to mis-operation of the evaporator liquid level.

4.9 Conclusions

The control of test R-64 was good except for lack of foresight in
prescheduling the evaporator liquid level control prior to the mid-test
calcination so that it would not overfill and cause the evaporator
conductivity to go high because of a safety override.

There was a total of 210 hr of controlled operation with only 38 times
that any of the 5 control functions was above the desired control limits.
The out of limit control averaged 7.6% of controlled operating time.
When the controlled signal did fluctuate the average deviation from the
set point was usually less than 1% and only the calciner liquid level
was greater, it was -3.2%.
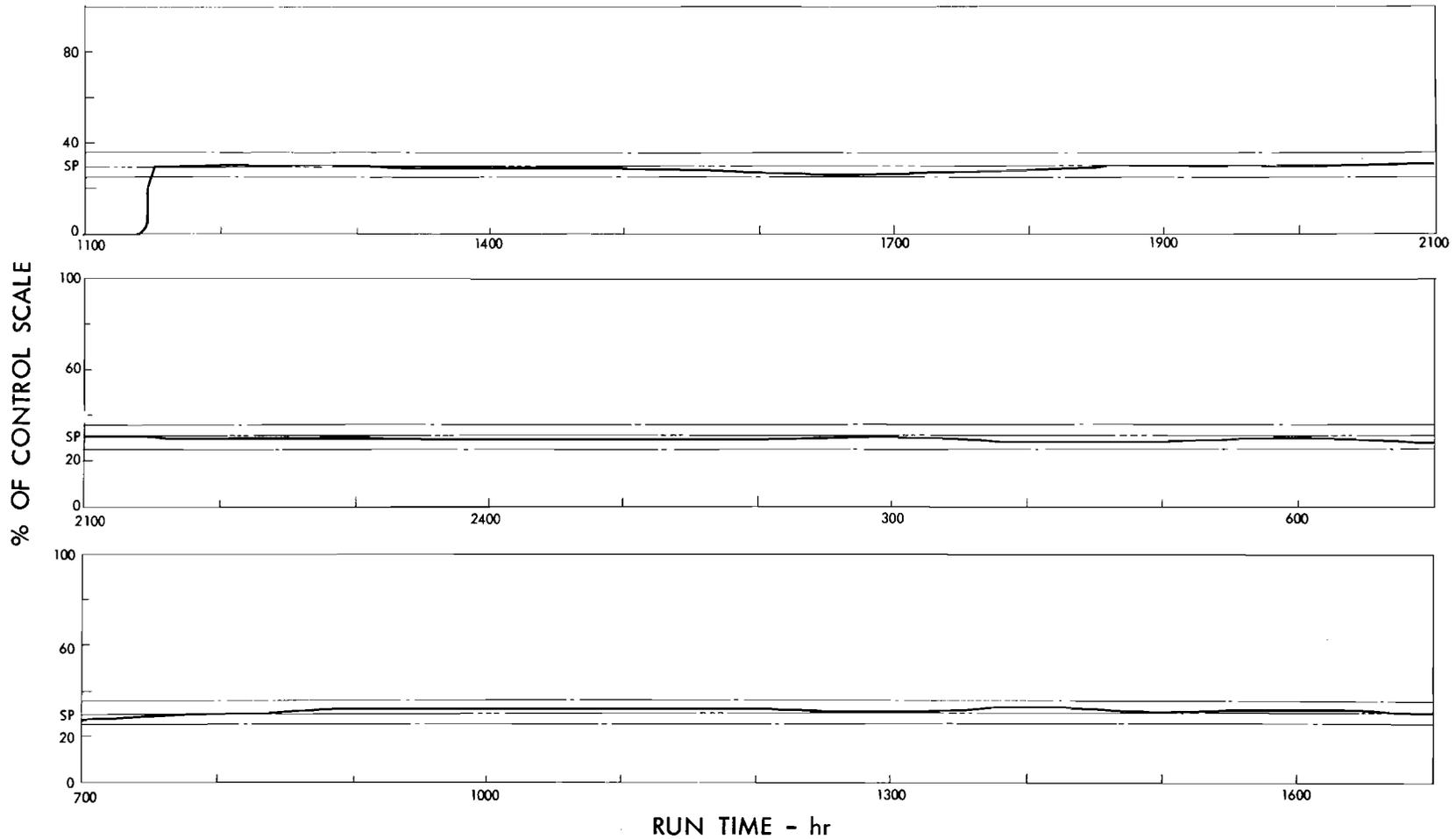
Fig. 4.4. Waste calcination & evaporation control – Test 64 – evaporator density.

Fig. 4.4. Waste calcination & evaporation control – Test 64 – evaporator density. (Cont'd)

Fig. 4.5. Waste calcination & evaporation control – Test 64 – evaporator vapor conductivity.

% OF CONTROL SCALE

80

SP 40

0
1700   2000   2300   200

80

SP 40

0
300   600   900   1200

RUN TIME - hr
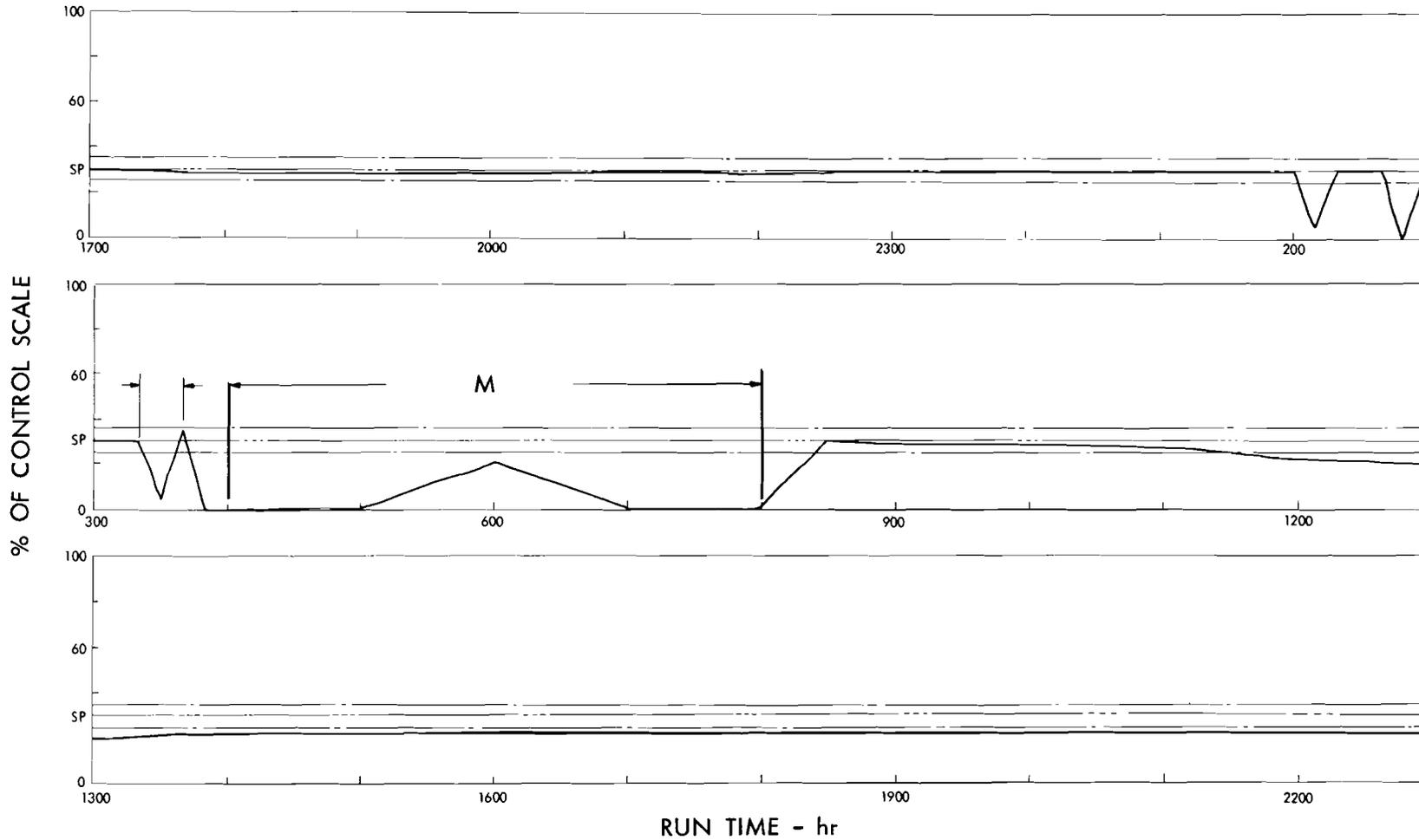
Evaporator Vapor Conductivity - Continuous - TBP
Limits SP ± 15% Controller Action   Prob. Band 200%
                               Reset   10 min

Fig. 4.5. Waste calcination & evaporation control - Test 64 - evaporator density. (Cont'd)

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