OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
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UNIT OPERATIONS SECTION MONTHLY PROGRESS REPORT

January 1962

CHEMICAL TECHNOLOGY DIVISION

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ABSTRACT

The experimental investigation of the reaction of H_2, CO, and CH_4 with CuO was extended to the range of concentration of 100 ppm or less of the contaminant in helium. The 250 ton prototype shear was dismantled for replacement of the stainless steel connecting rod with a chromium plated one equipped with a metal wiper. Inspection of the various shear components indicated negligible wear after ~1000 cuts on porcelain filled ORNL Mark I prototype fuel assemblies. The slip angle of sheared pieces in the leacher appears to be dependent on batch size and not on material type. HETS values for extraction and stripping using uranium as a stand-in for plutonium were determined as part of the amine extraction final cycle plutonium flowsheet studies. Installation of the 14-in.-ID rotary denitrator was completed and nine runs were made without mechanical difficulty but a creamy fraction and poor sol characteristics were observed. Installation of a weir in the aqueous leg of the Belgian mixer-settler between the mixer and settler resulted in better control of the aqueous-organic interface and organic backmixing.

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All previous reports in this series are listed in the June 1961 report, ORNL-TM-34, from the beginning, December 1954.

SUMMARY

1.0 GCR COOLANT PURIFICATION STUDIES

A gas-chromatographic technique was developed to give fast analysis of low concentrations of H_2 in helium with a sensitivity and accuracy of ~3 ppm in the range of 0 to 100 ppm.

The experimental investigation of the reaction of H_2, CO, and CH_4 with CuO was extended to the range of concentration of 100 ppm or less of the contaminant in the flowing stream of helium. Fair agreement was obtained between experimental results and predicted results of the previously assumed models.

2.0 POWER REACTOR FUEL PROCESSING

Shear and Leach. The development of a shear and leach complex consisting of a shear, conveyor-feeder, and leacher is continuing.

The stainless steel bellows in a 4-in. flapper valve failed at 35,400 cycles which is estimated to be equivalent to a service life of 4 years. Failure of the bellows does not hinder the mechanical operation of the valve.

The 250 ton prototype shear was dismantled for replacement of the stainless steel connecting rod with a chromium plated one equipped with a metal wiper to serve as a seal and prevent scoring of the connecting rod during shearing. Inspection of the various shear components indicated negligible wear after ~1000 cuts on porcelain filled ORNL Mark I prototype fuel assemblies.

The slip angle in the leacher for 1.5, 2.25, and 3.0 liter batches of empty 304L stainless steel tubing 1-in.-long, solid stainless steel rods (1/2-in. x 1-in. long), and sheared stainless steel clad porcelain 1 in. long varied from ~28-40 degrees at leacher elevations of 10, 20, and 30 degrees. The slip angle appears to be dependent on batch size and not on material type, and only slightly dependent on leacher elevation.

3.0 SOLVENT EXTRACTION STUDIES

HETS values for extraction and stripping using uranium as a stand-in for plutonium were determined as part of the amine extraction final cycle plutonium flowsheet studies. The uranium extraction HETS value for a sieve plate column (0.125-in.-dia holes, 23% free area, aqueous continuous operation) decreased from 6.6 to 4.2 ft as the pulse frequency increased from 50 to 70 cpm and for a nozzle plate column (0.125-in.-dia nozzles, 10% free area, solvent continuous operation), was 4.2 ft at a pulse frequency of 70 cpm. HETS values for uranium stripping decreased from 9.8 to 3.2 ft as the pulse frequency increased from 50 to 90 cpm for aqueous continuous operation of a sieve plate column and was 3.0 ft for solvent continuous operation of a nozzle plate column at 50 cpm pulse frequency.

4.0 THORIUM UTILIZATION STUDIES

Installation of the 14-in.-ID rotary denitrator was completed and nine runs were made without mechanical difficulty using 30 kg charges of $Th(NO_3)_4$ crystals. A creamy fraction and poor sol characteristics were present to a high degree for RD-1 product, but decreased for subsequent runs until RDB-9 and RDB-10 products dispersed into excellent sols. Contamination of the $Th(NO_3)_4$ or the steam and the slow denitration rate of the initial runs were eliminated as primary causes of the creamy fraction. Organic contamination of the denitrator or inadequate steam-charge contact remain as likely possible causes. Carryover of less than 1 per cent of the Th charge, run times of about six hours, and insignificant metal contamination of the product were demonstrated.

5.0 TRANSURANIC STUDIES

Stage efficiency of the Belgian mixer-settler was improved from ~60% to ~90% by the installation of 4 blade Teflon or stainless steel impellers. Design and installation of a weir in the aqueous leg between the mixer and settler resulted in better control of the aqueous-organic interface and markedly reduced the possibility of organic backmixing. The magnitude of the aqueous/organic flow ratio (A/O) greatly affects the stage efficiency from ~45% at an A/O of 8.4 to ~90% at an A/O of one.

1.0 GCR COOLANT PURIFICATION STUDIES

J. C. Suddath

1.1 Development of Chromatographic Techniques for Analysis of Hydrogen in Helium - C. D. Scott

Previously, chromatographic methods for analysis of H_2 in helium with co-contamination by H_2O, CO, CO_2, and CH_4 were worked out.[1,2] These methods were adequate for H_2 concentrations in the range of 100-20,000 ppm (vol) with analytical hydrogen sensitivity of 10-20 ppm. Although H_2 analysis by mass spectroscopy can be made with sensitivity and accuracy of ~1 ppm routine mass spectroscopy methods gave very uncertain results at H_2 concentration of less than 10 ppm.

In order to have fast, routine analysis of low concentration H_2 in helium at the experimental site, a gas-chromatographic technique was developed which was apparently capable of analyzing for H_2 with a sensitivity and accuracy of ~3 ppm in the range of 0-100 ppm H_2.

This technique involved separation of the H_2 and He by use of a 100-ft long, 1/4-in.-OD sorption column containing type 5-A Molecular Sieves in the size range of 18-30 mesh. This sorption column was used in the existing chromatograph which is a component of the GCR Coolant Purification Test Facility.[1]

It was found that with an argon carrier and reference gas at rates of ~50 cc/min and 25 cc/min, respectively, a 1.0 cc injection of the He-H_2 mixture resulted in a helium peak at the conductivity cell detector in approximately 8 min and a H_2 peak in approximately 10-1/2 min which allowed sufficient separation for detecting the two components. The chromatograph was calibrated with standard mixtures of H_2-He (determined by mass spectroscopy) containing 97 ppm, 57 ppm, 30 ppm, and 8 ppm H_2, and the resulting chromatogram was found to have linear response in this concentration range with a minimum detectable limit of 3 ppm (assuming linear response down to this level) and an accuracy, due to interpolation on the chromatogram, of approximately 3 ppm. There was sufficient uncertainty in the value of the H_2 concentration of the standard mixture ($\pm$ 5%) that the actual accuracy of the chromatography technique could not be established any closer than 5%.

1.2 Kinetics of Reaction of H_2, CO, and CH_4 with CuO at Low Concentrations

It is desirable to extend the experimental investigation of the reaction of H_2, CO, and CH_4 in a flowing stream of helium with fixed beds of CuO pellets to low concentrations of H_2, CO, CH_4 (< 100 ppm). A series of differential bed tests and deep bed tests were made in which He contamination with H_2, CO, and CH_4 in the range of ~100 ppm or less were used.

1.3 Differential Bed Tests

A series of 6 differential bed tests were made with the same experimental procedure previously used.[2] All of these tests were made at

500°C ± 10°C, 20.4 atm pressure, and with contaminant concentrations of 8-123 ppm (vol) (Table 1.1). One thermal balance run was made in which CH_4 in helium at a concentration of 137 ppm was passed over a bed of CuO pellets in a thermal balance[3] at 500°C and atm pressure. Operating procedures were similar to those previously used.[3]

The physical appearance of the sectioned CuO pellets from the differential bed tests with H_2 and CO was similar to that in the higher concentration tests, that is, there was a definite Cu-CuO reaction interface in the sectioned pellet. It was possible to obtain values of the effective porosity from these tests using the same method as outlined in a previous report.[4] These values of effective porosity were in the same range as the values determined using H_2 and CO concentrations in the range of 0.071 to 1.08 vol % (Table 1.2).

1.4 Deep Bed Tests

A series of deep bed tests were also made in which H_2, CO, and CH_4 in He at concentrations of ~100 ppm were allowed to react with fixed beds of CuO pellets. Experimental procedures were similar to those used earlier.[2] All tests were made in the 2-in.-dia oxidizing vessels in the GCR Coolant Purification Test Facility[1] at temperatures of 500 ± 15°C, 20.4 atm pressure, helium flow rates of 42 and 68 slpm and bed depths of 2-6 in. (Table 1.3). Analysis of the oxidizer effluent was by gas-adsorption chromatography. Initial results indicate fair agreement between experimental results and predicted results from the earlier assumed physical and mathematical models.

References

1. Unit Operations Monthly Progress Report, February 1960, ORNL CF 60-2-56.

2. Unit Operations Monthly Progress Report, March 1961, ORNL CF 61-3-67.

3. Unit Operations Monthly Progress Report, September 1961, ORNL-TM-112.

4. Unit Operations Monthly Progress Report, June 1961, ORNL-TM-34.

Table 1.1. Operating Conditions for Differential Bed Tests Made With Differential Beds of CuO Pellets at 500°C in Helium Gas Streams Containing, H_2, CO, or CH_4

Test No.	Contaminant	Average Contaminant Concentration, ppm	Length of Test, hrs
X-1	H_2	19	7
X-2	H_2	98	10
X-3	H_2	8	60
X-4	CO	123	10
X-5	CO	29	14
X-6	CH_4	115	8
X-7*	CH_4	137	12

* All tests were made at 20.4 atm except for X-7 which was a thermal-balance test at atm pressure.

Table 1.2. Effective Porosity of CuO Pellets for Mass Transfer of H_2 and CO as Determined from Differential Bed Tests of the H_2-CuO and CO-CuO Reactions at 500°C and 20.4 atm

Run No.	Contaminant	Contaminant Concentration, ppm	Effective Porosity
X-1	H_2	19	0.0672
X-2	H_2	98	0.0578
X-3	H_2	8	0.0552
X-4	CO	123	0.0573
X-5	CO	29	0.0519

Average effective porosity determined from previous tests = 0.0560.

Table 1.3. Experimental Conditions for Deep Bed Tests on the Kinetics of the Reactions of Low Concentrations of H_2, CO, and CH_4 in a Flowing Stream of Helium with Fixed Beds of CuO Pellets at 500°C and 20.4 atm

Run No.	Bed Depth in 2-in.-dia Reactor, in.	Contaminant	Average Contaminant Concentration in Feed Gas, ppm	Helium Flow Rate, slpm	Length of Test, hrs
Z-1	2.0	H_2	110	42	14
Z-2	6.0	H_2	127	68	60
Z-3	2.0	CO	142	42	16
Z-4	6.0	CH_4	94	42	8
Z-5	2.0	H_2 and CO	155 and 158	68	100

2.0 POWER REACTOR FUEL PROCESSING

C. D. Watson

2.1 Shear and Leach - B. C. Finney, G. A. West

A shear and leach program to determine the economic and technological feasibility of leaching the core material (UO_2 or UO_2-ThO_2) from relatively short sections (1-in. long) of fuel elements produced by shearing is continuing. This processing method enjoys the apparent advantage of recovering fissile and fertile material from spent power reactor fuel elements without dissolution of the inert jacketing and end adaptors. These unfueled portions are stored directly in a minimum volume as a solid waste. A "cold" shear and leach complex consisting of a shear, conveyor-feeder, and leacher is being evaluated prior to hot runs.

Life Testing of a 4-in. Bellows Sealed Flapper Valve. A 3-in.-ID x 3-3/32-in.-OD stainless steel bellows of 7 convolutions rolled from 5 mil thick tubing sealing the actuating arm of a 304-L stainless steel 4-in. flapper valve (Figures 2.1 and 2.2) failed at 35,400 operational cycles of opening and closing. Failure of the bellows allows air to leak into the valve but does not interfere with proper mechanical operation. A similar stainless steel bellows tested outside the valve failed at 50,000 cycles. Assuming one cycle per hour, the service life of the bellows is estimated to be 4 years. The flapper valve is being developed for use between the shear and conveyor-feeder and on the solids discharge port of the leacher.

Shear. The 250 ton prototype shear was dismantled to replace the stainless steel connecting rod with a chromium plated one containing a seal ring to dislodge particulates and prevent scoring during shearing. The fuel assembly stop which sets the length of the sheared fuel was replaced with a modified stop so that the length of cut can be varied from 1/4 to 1-1/2 inches.

Inspection of the shear components, fixed and moving blades, bearing surfaces of ram and gibs which determines the clearance between fixed and moving blades showed negligible change from original specifications after shearing ORNL Mark I assemblies 1,000 times. Scored places on the gibs and liners were cleaned up by grinding and shims were used to retain original clearances. A slight misalignment between the connecting rod and ram on a horizontal plane caused the ram to be in a "cocked" position during operation which prevented proper adjustment of the clearance between the moving and fixed blades. The rear of the ram in the area covered by the connecting rod was ground to allow more freedom of movement between the ram and connecting rod thus permitting the proper adjustment between blades.

Proximity switches with the appropriate control wiring were installed on the feed mechanism pusher arm hydraulic cylinder. These switches regulate batch size automatically by controlling the total length of fuel assembly sheared.

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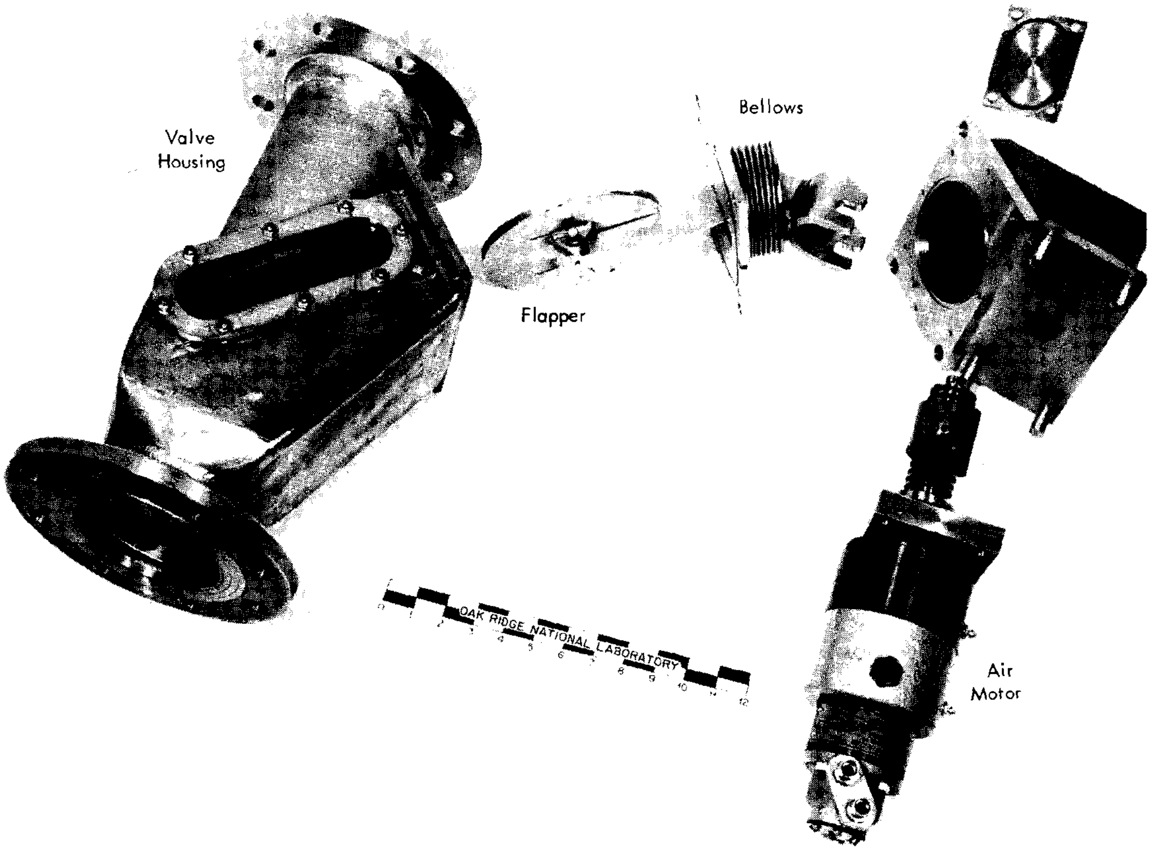

Fig. 2.1. Disassembled view of 4-inch bellows sealed flapper valve.

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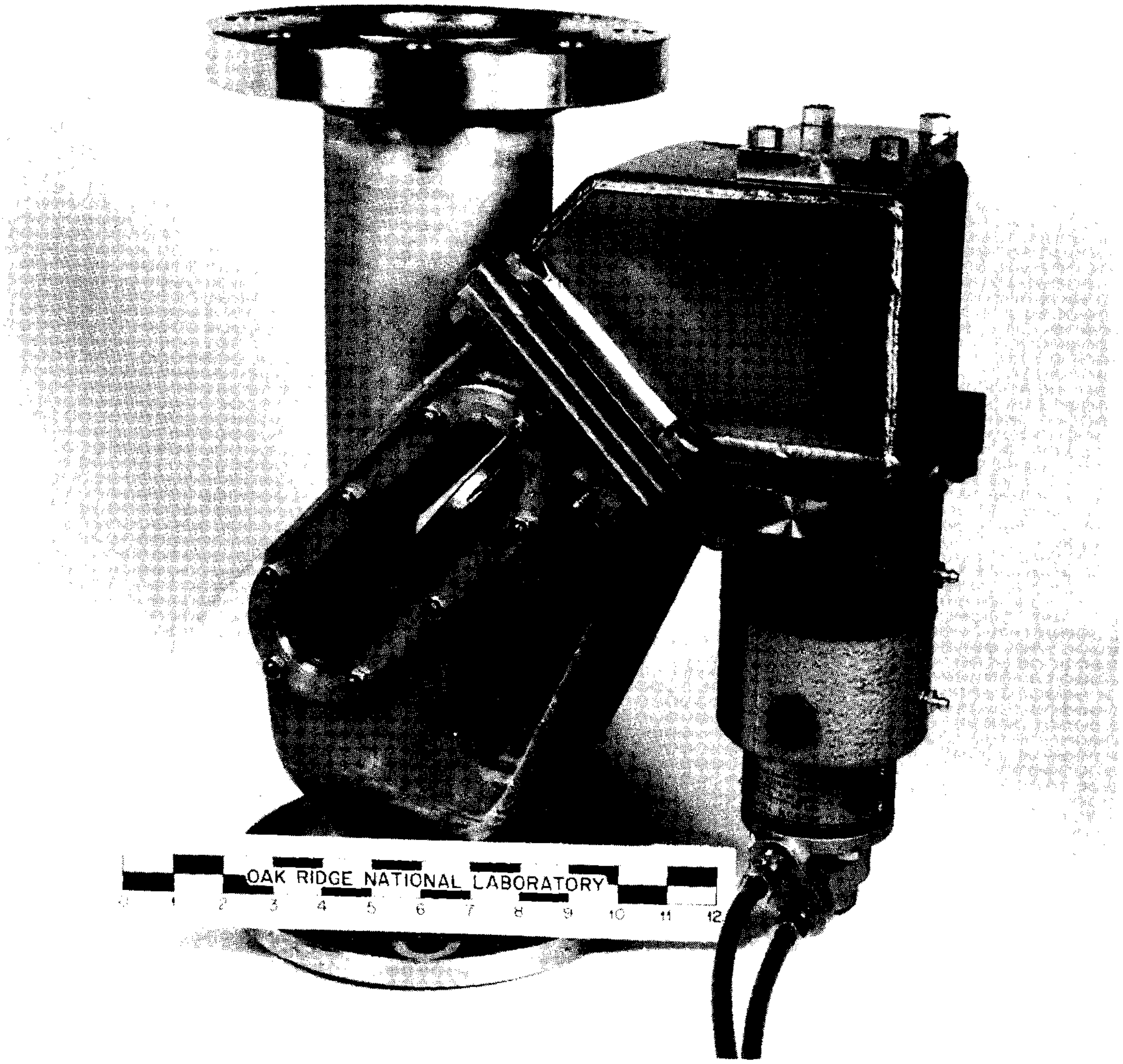
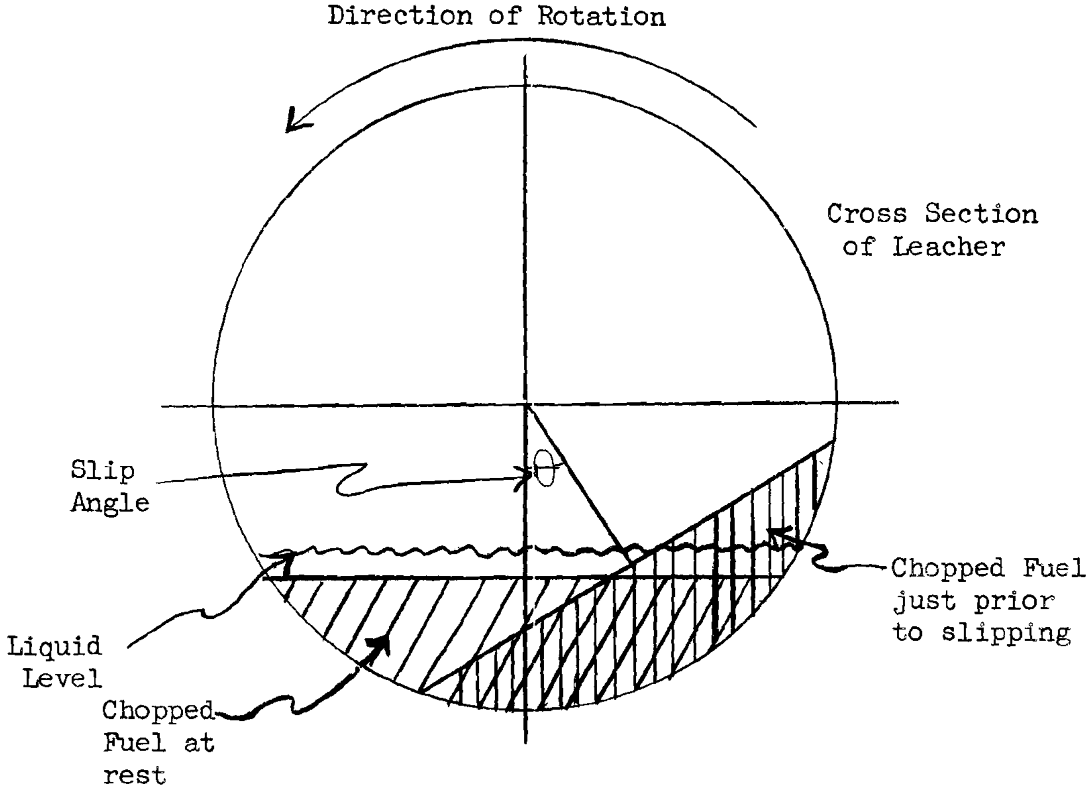


Fig. 2.2. Assembled view of 4-inch bellows sealed flapper valve.

Leacher. To insure submergence of sheared fuel under the acid dissolvent, the slip angle for 1.5, 2.25 and 3.0 liter batches of empty 304L stainless steel tubing (1-in. long), solid stainless steel rod (1/2-in.-dia x 1-in.-long) and stainless steel jacketed porcelain filled Mark I sheared 1-in.-long were measured at leacher elevations of 10, 20, and 30 degrees (Table 2.1). There appeared to be little difference in performance of equal volumes of empty cladding, solid stainless steel rods and porcelain filled Mark I elements. The angle of slip generally increased with batch volume and less significantly with leacher elevation. At a leacher elevation of 15°, the slip angle increased from about 28 to 32° when the batch volume increased from 1.5 to 3 liters. The optimum leacher batch size is 2.25 liters of sheared Mark I prototype which is equivalent to 8 in. of length.

The slip angle is defined as the counterclockwise angle of rotation of the leacher (looking into feed end of leacher) just sufficient to bring an originally horizontal batch to the point of slippage. (see diagram)



For the batch sizes studied, slippage first occurs when approximately one half of the batch is above the liquid level. To insure submergence of chopped fuel as it is advanced each flight, the leacher will have to be rotated 360° plus the slip angle (~28° for 2.25 liter batch) then reversed by the amount of the slip angle.

The feed inlet line to the leacher has been changed from the left side of the center shaft (looking at feed end of leacher) to the right side of the center shaft. This was necessary so that the leacher could be loaded with the beginning of the first flight at the 6 o'clock position.

The washing portion of the spiral inclined rotary leacher which was designed for continuous rotation of the unit directs the wash water less efficiently when the leacher is operated only once per hour. To prevent wash water from being discharged (with the leached shells) the wash section flights were perforated so that wash water would flow countercurrent flight to flight. Because of the dumping characteristics of the perforations, the holdup of water in the washing section when the leacher makes a complete revolution varies from 0-3 liter as the flow rate increases from 0-5 liters/time cycle. A flow rate in excess of 5 liters/time cycle results in water being discharged from the leacher through the dissolution section dump screen.

Table 2.1. Slip Angle of Materials in Transport in Leacher

Material	Batch Size liters	Weight, kilograms	Slip Angle Leacher Elevation, degrees 10	15	20
Empty 304L SS tubing	1.5	1.7	24	31	28
Empty 304L SS tubing	2.25	2.5	28	31	32
Empty 304L SS tubing	3.00	3.3	34	36	40
Solid SS rod (1/2-in.-dia x 1-in.-long)	1.5	6.7	27	28	36
Solid SS rod (1/2-in.-dia x 1-in.-long)	2.25	9.7	34	33	34
Solid SS rod (1/2-in.-dia x 1-in.-long)	3.00	12.9	34	32	40
Stainless steel jacketed porcelain filled Mark I sheared 1-in. long	1.5	2.4	27	28	28
Stainless steel jacketed porcelain filled Mark I sheared 1-in. long	2.25	3.6	26	32	37
Stainless steel jacketed porcelain filled Mark I sheared 1-in. long	3.00	4.8	31	30	39

3.0 SOLVENT EXTRACTION STUDIES

A. D. Ryon

The HETS values reported this month were obtained as part of the amine extraction final cycle plutonium recovery flowsheet studies.

3.1 Pulse Column Efficiency Studies - R. S. Lowrie

Lack of containment precluded pulse column efficiency studies with plutonium under amine extraction final cycle plutonium flowsheet conditions; so uranium was used as a stand-in. This necessitated using a mixed nitric acid-aluminum nitrate salted feed to obtain an extraction coefficient for uranium that would permit column operation at the same flow ratio as in the plutonium flowsheet. Similarly, 0.2 M nitric acid was used as the stripping agent since its coefficient permitted operation at the desired 1/5 A/O phase ratio. The uranium test flowsheet is shown in Figure 3.1. The compositions and physical properties of the various entering streams and equilibrated solvent-aqueous pairs typical of the top and bottom of both extraction and stripping columns are shown in Table 3.1. The increase in solvent viscosity which occurred in the bottom of the extraction column as the free amine converted to the acid salt was the only major change in the physical properties.

Two arbitrary rules were used in the HETS calculations, first, calculations were based on the analyses of the phase from which the uranium transferred, i.e. aqueous phase for extraction, solvent phase for stripping, and second, the continuous phase profile samples were used in calculating incremental HETS values since the differential samplers operated in such a manner that these samples were most representative of conditions actually present in the column. This, in turn, required calculating by material balance the aqueous profile concentrations for solvent continuous operation of the extraction column and solvent profile concentration for aqueous continuous operation of the stripping column to satisfy the first condition.

3.2 HETS Values for Uranium Extraction

Uranium extraction efficiencies were determined for aqueous continuous operation of a sieve plate column (0.125-in.-dia holes, 23% free area, 24 ft long) and for solvent continuous operation of a nozzle plate column (0.125-in.-dia nozzles, 10% free area, 24 ft long). Actual operation conditions and column profile sample analyses are shown in Table 3.2, the calculated HETS values in Table 3.3, and a typical McCabe-Thiele diagram in Figure 3.2. HETS values for uranium extraction increased from 4.2 ft to 6.6 ft (average of runs 19-27 and 23-24) as pulse frequency decreased from 90 to 70 cpm for aqueous continuous operation of the sieve plate columns. Solvent continuous operation of the nozzle plate column at 70 cpm resulted in a HETS value of 4.2 ft for uranium extraction.

In all cases, the actual profile analyses plotted either on or very close to the operating line indicating that no significant extraction occurred during the sampling procedure and, further, that no significant

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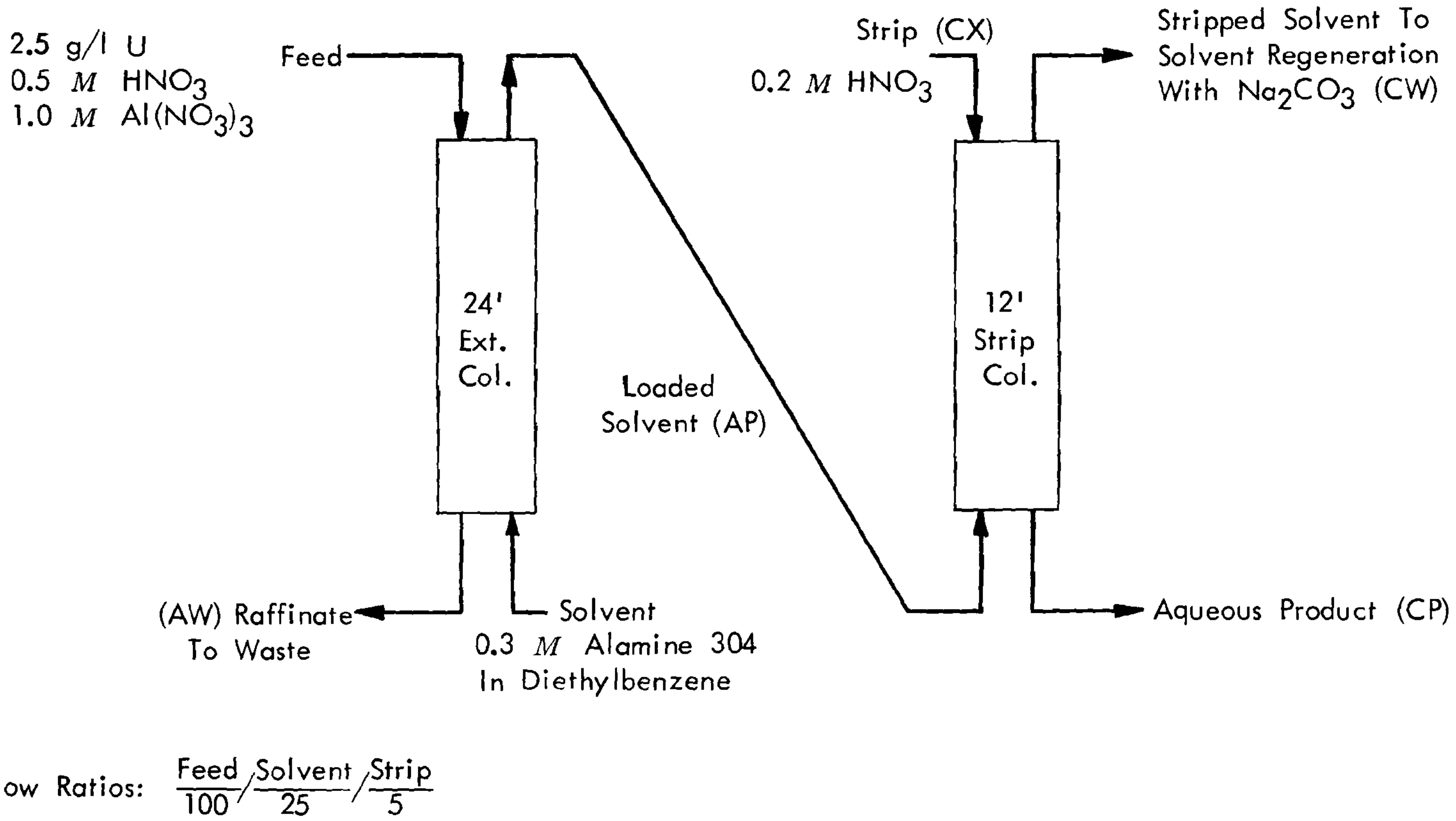

Fig. 3.1. Flowsheet for pulse column efficiency tests.

Table 3.1. Properties of Test Solutions (25°C)

Test Solution	U g/ℓ	H^+ M	Viscosity centipoise	Density g/cc	Interfacial Tension dynes/cm
Feed	2.5	0.5	2.02	1.18	
Solvent*	0	0	1.29	0.85	
Strip	-	0.2	0.86	1.01	
Extraction Column					
Top (Solvent	8.39	0.45	2.70	0.88	19.6
End (Aqueous	1.19	0.54	2.04	1.18	
Bottom (Solvent	0.07	0.45	2.58	0.86	20.1
End (Aqueous	0.001	0.42	2.05	1.18	
Strip Column					
Bottom (Solvent	5.67	0.34	2.70	0.87	19.1
End (Aqueous	32.8	0.52	0.93	1.06	
Top (Solvent	< 0.001	0.34	2.49	0.87	19.2
End (Aqueous	0.005	0.34	0.85	1.01	

* Measured as free amine.

Table 3.2. Experimental Conditions and Column Profile Samples - Extraction

Run No.	19-27				23-24				23-2				19-38A				
Column* Cartridge	Sieve				Sieve				Sieve				Nozzle				
Cont. Phase	Aq				Aq				Aq				Solv				
Pulse Frequency, cpm	70				70				90				70				
U Material Balance, %	105.5				100.9				102.2				104.8				
	Aqueous		Solvent		Aqueous		Solvent		Aqueous		Solvent		Aqueous			Solvent	
Flow Rate gal $ft^{-2}hr^{-1}$	720		200		730		190		380		90		365			95	
Location of Profile Samples Feet from Top	U g/ℓ	H^+ M	U g/ℓ	H^+ M	U g/ℓ	H^+ M	U g/ℓ	H^+ M	U g/ℓ	H^+ M	U g/ℓ	H^+ M	U g/ℓ	U** g/ℓ	H^+ M	U g/ℓ	H^+ M
AP-Feed	2.74	0.50	9.2	0.4	2.71	0.50	10.4	0.40	2.80	0.52	11.6	0.40	2.50	2.57	0.45	9.75	0.39
2	2.36	0.51	8.20	0.39	2.29	0.48	8.52	0.43	2.21	0.52	9.11	0.42	1.96	2.16	0.51	8.20	0.39
4	2.11	0.51	7.20	0.39	2.02	0.48	7.22	0.43	1.78	0.51	7.64	0.40	1.34	1.70	0.51	6.46	0.39
6	1.79	0.51	6.06	0.39	1.66	0.48	5.79	0.43	1.28	0.51	5.59	0.42	1.06	1.22	0.51	4.63	0.40
8	1.52	0.51	5.03	0.39	1.32	0.48	4.75	0.42	0.87	0.51	3.82	0.43	0.68	0.86	0.51	3.23	0.40
10	1.36	0.51	4.36	0.39	0.97	0.48	3.60	0.43	0.56	0.52	2.33	0.43	0.53	0.58	0.51	2.17	0.41
12	1.08	0.51	3.19	0.39	0.70	0.48	2.51	0.44	0.37	0.51	1.44	0.43	0.29	0.39	0.51	1.46	0.41
14	0.88	0.51	2.46	0.39	0.48	0.48	1.80	0.43	0.20	0.51	0.85	0.43	0.20	0.27	0.51	0.96	0.41
16	0.69	0.50	1.74	0.37	0.31	0.48	1.16	0.42	0.10	0.51	0.51	0.41	0.10	0.16	0.51	0.60	0.41
18	0.53	0.50	1.23	0.35	0.21	0.47	0.74	0.39	0.06	0.51	0.36	0.42	0.08	0.10	0.51	0.37	0.41
20	0.41	0.49	0.76	0.32	0.14	0.46	0.54	0.38	0.03	0.51	0.15	0.38	0.031	0.06	0.49	0.29	0.41
22	0.29	0.47	0.34	0.26	0.09	0.44	0.25	0.28	0.02	0.48	0.116	0.31	0.024	0.039	0.48	0.13	0.40
24	0.21	0.42	0.05	0.12	0.06	0.38	0.07	0.09	0.011	0.41	0.040	0.07	0.015	0.030	0.42	0.09	0.30
Aw-Solv.	0.20	0.40	0.01	0.00	0.04	0.20	0.001	0.00	0.011	0.37	0.040	0.00	0.015	0.015	0.38	0.03	0.00

* Sieve plate - 0.125-in.-dia holes, 23% free area; nozzle plate - 0.125-in.-dia holes, 10% free area.
** Calculated by material balance from solvent.

Table 3.3. Calculated HETS Values for Uranium Extraction

Location of Column Sections Feet from Top	HETS Values - Feet			
	Run No. 19-27	Run No. 23-24	Run No. 22-2	Run No. 19-38
0-2	5.4	6.1	3.5	4.0
0-4	6.3	7.4	4.0	3.5
0-6	6.1	7.2	3.9	3.4
0-8	6.3	7.0	4.1	3.4
0-10	6.8	6.5	3.9	3.5
0-12	6.8	6.5	4.2	3.6
0-14	7.1	6.2	3.9	3.7
0-16	7.0	6.0	4.1	3.8
0-18	7.0	6.2	4.1	3.9
0-20	7.2	6.1	4.1	4.1
0-22	7.4	6.5	4.2	4.2
0-24	7.2	6.0	4.2	4.1
2-4	7.7	8.3	4.7	3.4
4-6	6.3	6.1	3.8	3.1
6-8	6.7	5.7	3.6	3.3
8-10	9.0	4.6	3.6	3.4
10-12	5.7	4.9	2.9	3.6
12-14	8.0	4.3	2.9	4.0
14-16	6.3	3.8	2.2	3.3
16-18	6.5	4.6	3.4	4.4
18-20	6.9	4.0	3.2	4.8
20-22	5.7	4.7	4.8	4.0
22-24	6.7	3.7	3.1	6.7

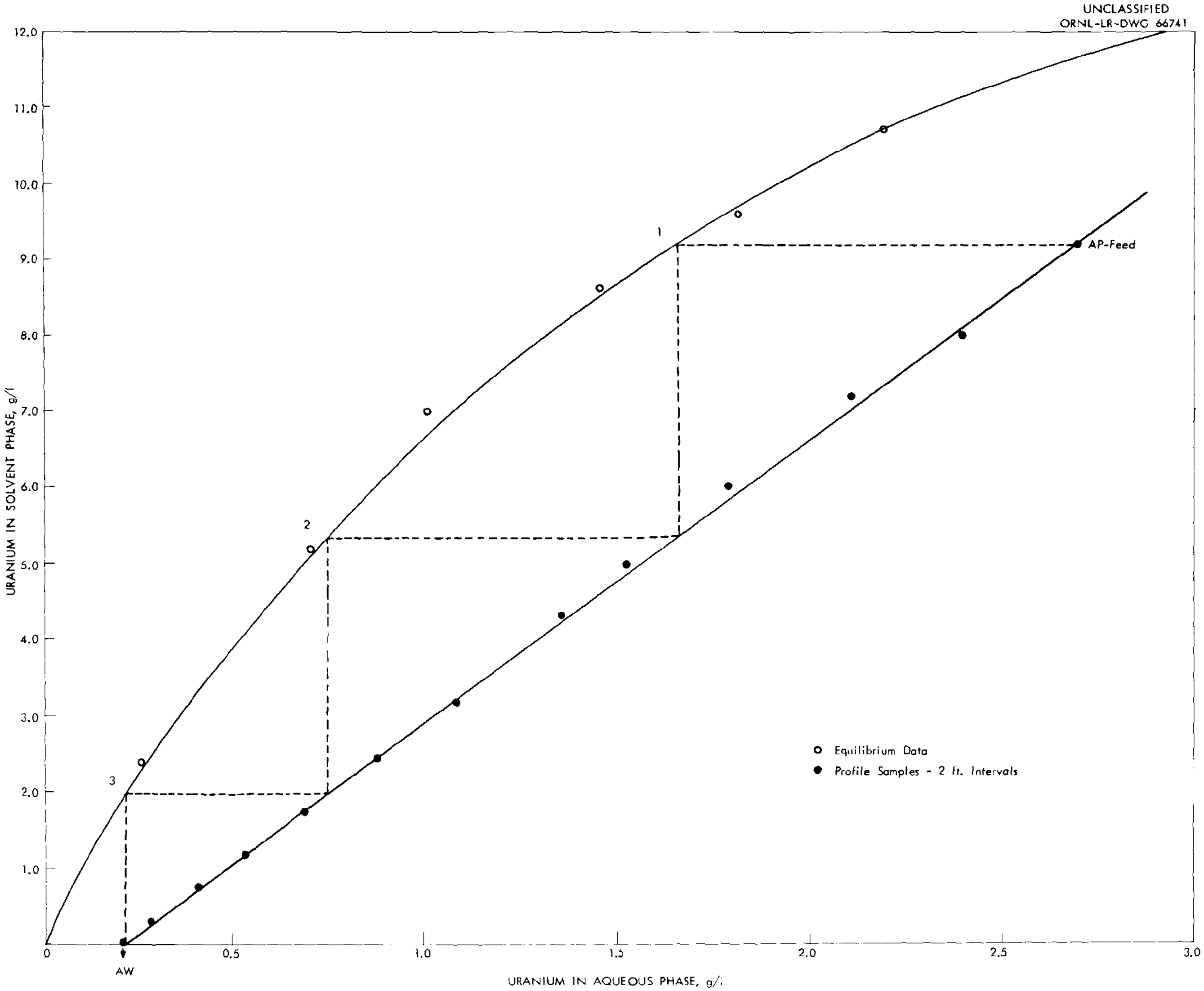

Fig. 3.2. McCabe-Thiele diagram for uranium extraction - Run 19-27.

backmixing occurred in the columns. Incremental HETS values, whether calculated for individual 2 ft column sections or over a progressively increasing column length were quite consistent showing that the extraction efficiency was constant over the entire length of the column.

3.3 HETS Values for Uranium Stripping

HETS values for uranium stripping were determined in 12 ft pulse columns equipped with either sieve or nozzle plates. The experimental conditions and column profile sample analyses are shown in Table 3.4. Since amine solvents readily extract both nitric acid and uranium, the aqueous nitric acid concentration varied markedly over the column, necessitating the use of "floating" equilibrium lines in McCabe-Thiele plot (Figure 3.3). The HETS values for uranium stripping in a sieve plate column operated aqueous continuous decreased from 9.8 to 3.2 ft as the pulse frequency increased from 50 to 90 cpm (Table 3.5). Solvent continuous operation of a nozzle plate column gave a value of 3.0 ft. Note that the incremental HETS values were quite constant.

Table 3.4. Experimental Conditions and Column Profile Samples - Stripping

Run No.	19-27					23-24					19-38B			
Col. Cartridge*	Sieve					Sieve					Nozzle			
Phase Cont.	Aq					Aq					Solv			
Pulse Frequency, cpm	50					90					50			
U Material Balance, %	102.7					94.1					101.7			
	Solvent			Aqueous		Solvent			Aqueous		Solvent		Aqueous	
Flow Rates, gal ft^{-2}hr^{-1}	213			50		204			42		96		22	
Location of Sample Points Feet from Bottom	U g/ℓ	U** g/ℓ	H$^+$ M	U g/ℓ	H$^+$ M	U g/ℓ	U** g/ℓ	H$^+$ M	U g/ℓ	H$^+$ M	U g/ℓ	H$^+$ M	U g/ℓ	H$^+$ M
AP-CP	9.2		0.40	25.4	0.43	10.4		0.4	37.3	0.60	7.03	0.39	28.5	0.50
2											5.76	0.35	26.3	0.34
4											4.25	0.34	21.6	0.31
6	6.03	6.03	0.36	10.1	0.31	7.15	7.65	0.37	27.5	0.38	2.95	0.33	14.9	0.25
8											1.86	0.32	8.06	0.21
10											1.09	0.32	4.79	0.20
CW-CX	3.55		0.33	0.0	0.20	2.03		0.34	0.0	0.20	0.74	0.32	0.0	0.20

* Sieve plate - 0.125-in.-dia holes, 23% free area; nozzle plate - 0.125-in.-dia, 10% free area.

** Calculated by material balances from aqueous samples.

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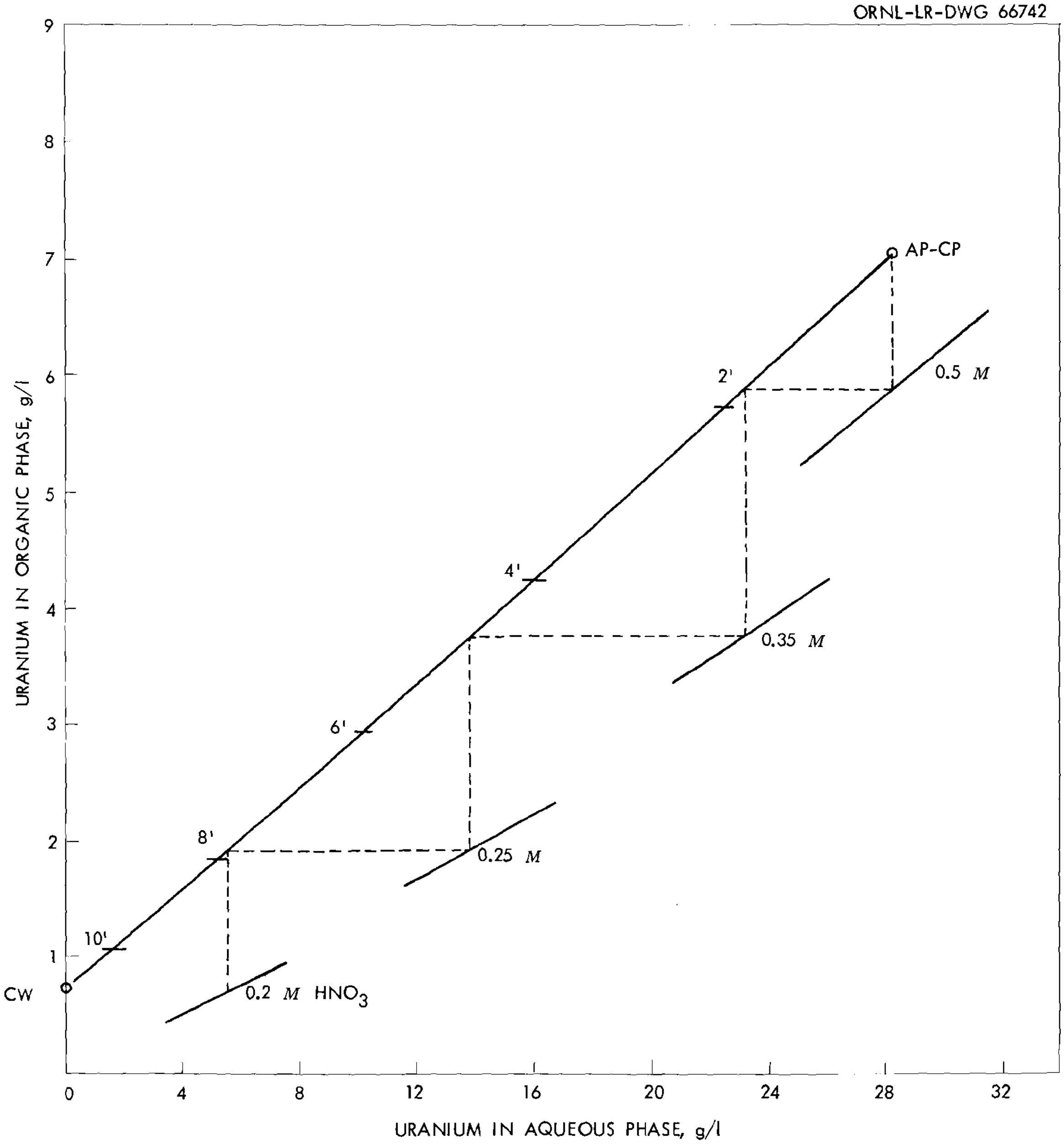

Fig. 3.3. McCabe-Thiele diagram for uranium stripping - Run 19-38B.

Table 3.5. HETS Values for Uranium Stripping

Location of Column Sections Feet from Bottom	HETS Values - Feet		
	Run No. 19-27	Run No. 23-24	Run No. 19-38B
0-2			1.9
0-4			2.2
0-6	8.6	3.3	2.6
6-12	11.6	3.2	-
0-8			2.7
0-10			2.7
0-12	9.8	3.2	3.0
2-4			2.6
4-6			2.7
6-8			2.9
8-10			3.1
10-12			3.9

4.0 THORIUM UTILIZATION STUDIES

P. A. Haas

This program is to develop chemical processing and fuel fabrication procedures applicable to use of thorium as a fertile material. The immediate objective is to develop to the pilot plant stage the application of the sol-gel and vibratory compaction processes to give clad oxide fuel elements. The Unit Operations studies are on denitration of $Th(NO_3)_4$ in steam atmospheres followed by dispersion into thoria sols, uranium addition, drying, calcination, and reduction to give high density ThO_2-UO_2 particles. Test elements of vibratorily compacted mixed oxides are being fabricated or irradiated. Individual steps of the sol-gel process are to be tested on a remote ThO_2-$U(233)O_2$ pilot plant scale.

4.1 Thorium Nitrate Denitration Studies - J. W. Snider, R. D. Arthur, D. A. McWhirter

Installation and shakedown of the 14-in. rotary denitrator was completed and the first series of runs began. The flowsheet of the rotary denitrator and auxiliary equipment is as shown in Figure 4.1. The sol storing and aging tanks are not yet installed.

A photograph of the rotary denitrator in the unloading position is shown in Figure 4.2. For unloading, a plastic sock was taped around a 4-in. pipe adaptor and extended into the receiving vessel. The denitrator was then rotated at ~2 rpm and tilted slowly into the position shown in Figure 4.2. This method of unloading proved very effective in preventing dusting inside the hood. For loading, the discharge end was elevated and thorium nitrate cyrstals fed through a funnel into the denitrator.

Thermocouples were installed on the drum to measure the skin temperature after run RD-1. Three thermocouples, 1/16-in.-OD-stainless sheath with MgO filling, were placed on the drum and one inside the steam supply line. One of the thermocouples failed when the leads shorted to the stainless steel sheath. This became a frequent problem with these thermocouples due to vibration at the commutator end. A special thermocouple end support is being fabricated to prevent this. In all runs, one of the two remaining drum thermocouples performed satisfactorily.

An inlet steam baffle, 4-in.-dia, was installed and used for the last two runs. The baffle is located in the enlarging inlet section such that ~1 in. of clearance exists between the baffle and drum conical section. A smaller 2-in.-dia baffle is located ~4 in. from the steam exit point. The purpose of the baffles was to deflect the steam and prevent channeling at the low flow rates and to thereby increase the steam velocity over the particles.

4.2 Rotary Denitrator Operating Conditions and Results

Nine runs were made with the rotary denitrator using a charge of 30.0 kg of thorium nitrate crystals and one run using 10.6 kg of thorium nitrate

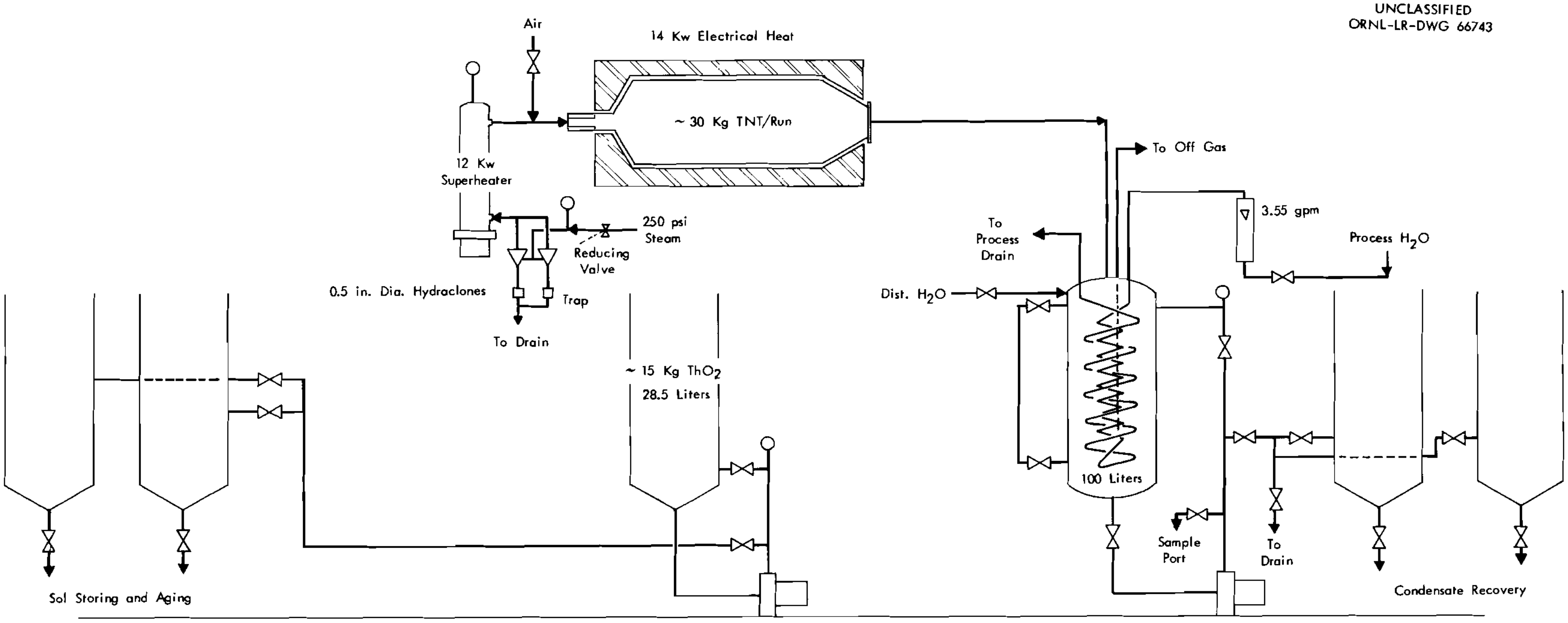

Fig. 4.1. 14-in. rotary denitrator and auxiliary equipment - flow sheet.

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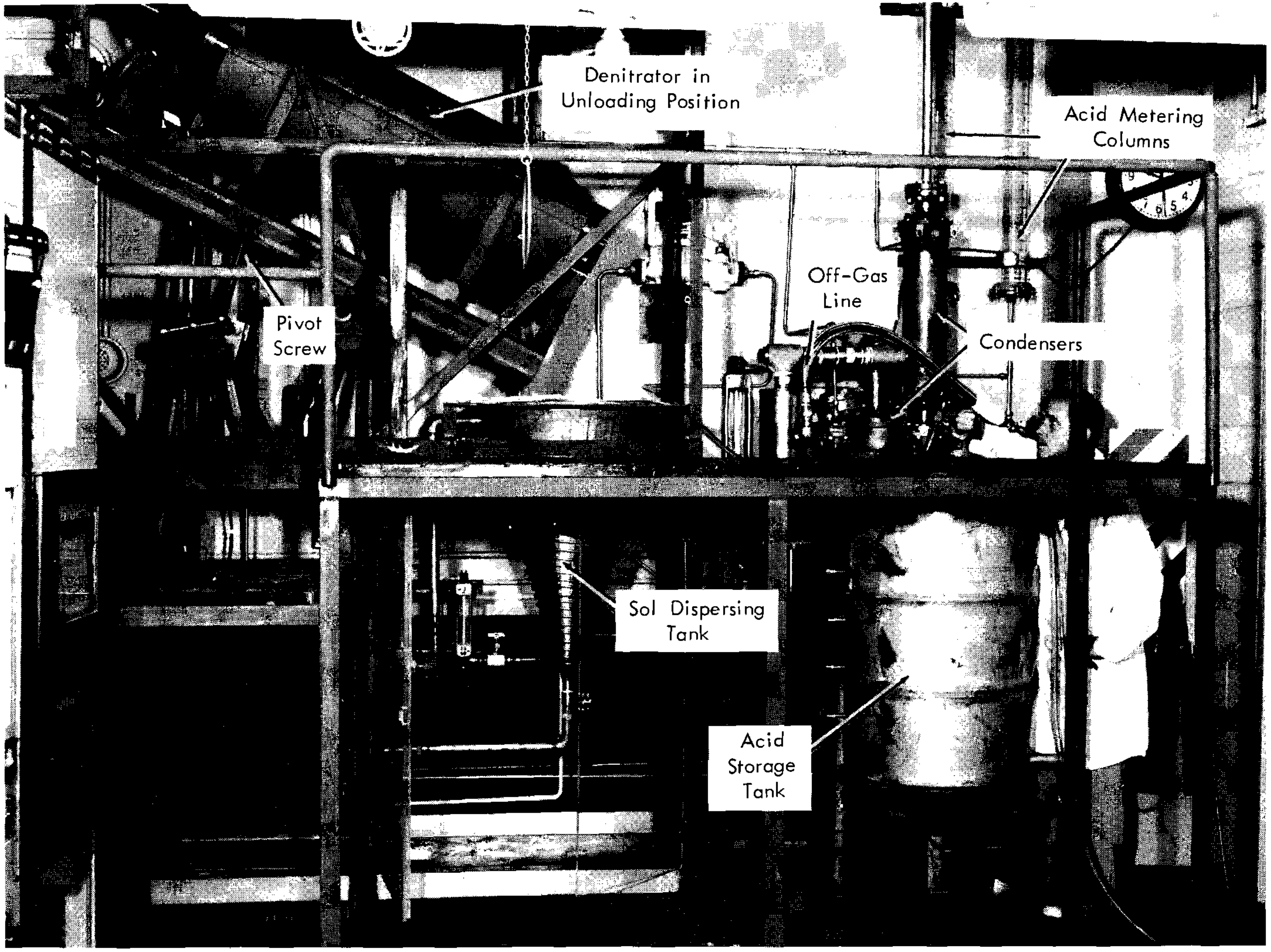

Fig. 4.2. Rotary denitrator in the unloading position.

crystals. Run conditions and the product analysis are tabulated in Tables 4.1 and 4.2. The percent of the feed nitrate collected in the off-gas condenser as a function of time is shown in Figures 4.3 and 4.4.

The rotating denitrator skin temperatures as a function of time for runs RD-2 through RD-10 are shown in Figures 4.5 and 4.6. In general, two hold periods were observed during which the temperature rise was interrupted even though the heaters remained on. They were at 225°C and 300°C. The lower hold period is believed to represent the heat of melting from crystal to syrup form. The 300°C hold is believed to represent the transition from a syrup into a granular powder. The evidence, other than the heat of reaction, which led to this conclusion was observed during run RD-3. During run RD-3 the stainless steel balls could be heard rattling around until the 225°C hold was reached. Between the 225°C and the 300°C holds a very muffled sound existed which was reduced to silence as the 300°C level was approached. Upon reaching the 300°C plateau, the calciner drive stalled and manual assistance by use of a pipe wrench for about two revolutions was required. During these assisted revolutions the balls could be heard dropping from considerable distances, as if the syrup held them to the drum wall until they were near the top. Operation then returned to normal with a noise similar to that noted prior to reaching the 225°C plateau.

4.3 Dispersion of Denitrator Products

The N/Th ratio of run RD-1, 0.127, was within the range of a stable sol; but the product would not disperse. This product analyzed similar to products produced with the agitated trough calciner in which steam was used for 135 min with no air. Iron contamination was somewhat higher due to first run cleaning, however, it was not high enough to account for the abnormal behavior of this product. The surface area, 23.2 in.^{2}/g, was approximately one-half that of all remaining runs made with the rotary denitrator. It does compare closely to the surface area of products produced in the agitated trough calciner in which steam was used for 3 hrs followed by air for 3 hrs (run ATC-55 product analyses are included to show this). A 2 kg lot of RD-1 product was charged to the agitated trough calciner and contacted with steam for 3 hrs. This reduced the N/Th ratio to 0.029. If this improved the quality of the product, it was only a marginal improvement. This material is labeled RD-1-ATC-3 in Table 4.2.

The product from run RD-1, although analyzing very nearly the same as the product from run 48 made in the agitated trough calciner (Table 4.2), contained a high per cent of material which appeared creamy; and all efforts to disperse the material by techniques used on agitated trough calciner materials failed. Reflux with concentrated nitric acid which was subsequently diluted out did disperse the material; however, this process dissolved ~20% of the thorium. The creamy fraction, ~80% of the product from run RD-1, appeared similar to one lot of material produced by Chemical Development Section A in the presence of organic impurities.

Table 4.1. Rotary Denitrator Run Conditions and Objectives

Charge: 30 kg of $Th(NO_3)_4 \cdot XH_2O$ or as Noted

Run No.	Heater Program Temp, °C	Heater Program No. of Hours	Steam Program Flow Rate lb/hr	Steam Program Inlet Temp, °C	Steam Program No. of Hours	Percent Thorium Carryover	Purpose of Run, Other Conditions and Remarks
1	400 425	6.66 3.50	22	-	7.50	-	Purpose: Initial run with system. Other Conditions: Air at 1.33 SCFM for 3.00 hr used at end of run.
2	425	8.00	40	350	8.00	9.75	Purpose: To increase heat flux into drum, and time of steam contact.
3	425	7.17	48 22	350 325	2.16 4.33	0.94	Purpose: To study the effect of pulverizing the powder while in contact with steam. Other Conditions: 19 kg of 5/8-in.-dia 316 stainless steel balls charged with $Th(NO_3)_4 \cdot XH_2O$.
4	460 475 485	3.25 0.58 2.50	40 22 11	385 350 255	2.00 1.17 3.25	1.53	Purpose: To study the effect of an increasing heat flux into the drum.
5	475	6.25	48 22 9	385 350 225	1.75 1.25 2.83	1.75	Purpose: To study the effect of a shorter run time.
6	475	5.75	48 22 9	390 350 225	1.33 1.00 3.17	1.75	Purpose: To study the effect of a small charge, and higher denitrating rate. Other Conditions: Charge was 10.59 kg of $Th(NO_3)_4 \cdot XH_2O$.
7	500	6.00	45 22 9	375 350 325	2.00 1.00 3.00	1.77	Purpose: To study the effect of increased heater temperature with a similar steam program as run 4.
8	500	10.00	45 22 9	375 350 325	1.00 2.00 3.00	0.82	Purpose: To study the effects of introducing air after denitrating to a low nitrate content, and a modified steam program's effect on the carryover. Other Conditions: Air at 1.33 SCFM for 4.00 hr used at end of run.
9	500	6.00	10-20	-	6.00	0.52	Purpose: To study the effect of an inlet steam baffle. Other Conditions: Steam controlled manually due to diaphragm rupture.
10	500	6.00	30 16	400 340	1.42 4.58	0.98	Purpose: Run 9 repeated to see if good product was reproducible.

Table 4.2. Analysis of Products Produced in the 14-in.-dia Rotary Denitrator

Run No.	RD-1	RD-1* ATC-3	RD-2	RD-3	RD-4	RD-5	ATC-55**	RD-6	RD-7	RD-8	RDB-9***	RDB-10
Time in steam, min	445	180	465	420	385	360	180	330	360	360	360	360
Time in air, min	180	-	-	-	-	-	180	-	-	240	-	-
Product weight, kg	14.61	-	12.47	14.20	13.86	13.72	-	4.63	13.87	14.44	14.02	13.65
Thorium carryover, %	-	-	9.75	0.94	1.53	1.75	-	1.75	1.77	0.82	0.52	0.98
LOI (300-1000°C), %	4.68	2.48	3.45	4.39	2.79	2.36	2.44	2.25	1.95	1.58	1.57	2.74
Nitrogen, %	0.65	0.15	0.40	0.56	0.21	0.19	0.24	0.08	0.17	0.16	0.15	0.20
Thorium, %	84.73	85.89	84.85	84.09	85.76	86.28	86.24	86.39	86.49	86.88	86.63	86.29
N/Th, mole ratio	0.127	0.029	0.078	0.110	0.041	0.035	0.046	0.015	0.033	0.031	0.029	0.038
Crystallite size, A°	59	-	66	65	77	67	66	78	78	80	70	73
Surface area, m^2/g	23.2	-	44.5	36.3	46.9	47.9	26.0	52.8	50.2	50.3	49.8	46.2
Fe, ppm	215	244	54	145	22	< 50	< 50	53	< 45	-	-	-
Cr, ppm	< 25	< 25	30	< 35	< 35	< 50	< 50	< 50	< 50	-	-	-
Cu, ppm	< 50	< 50	< 70	< 75	< 75	< 50	< 50	< 50	< 50	-	-	-

* Product from run RD-1 charged into agitated trough calciner and contacted with steam for 3 hr.

** Run ATC-55, agitated trough calciner, was made on the same day using feed from the same drum as run RD-5.

*** "B" signifies baffled steam inlet.

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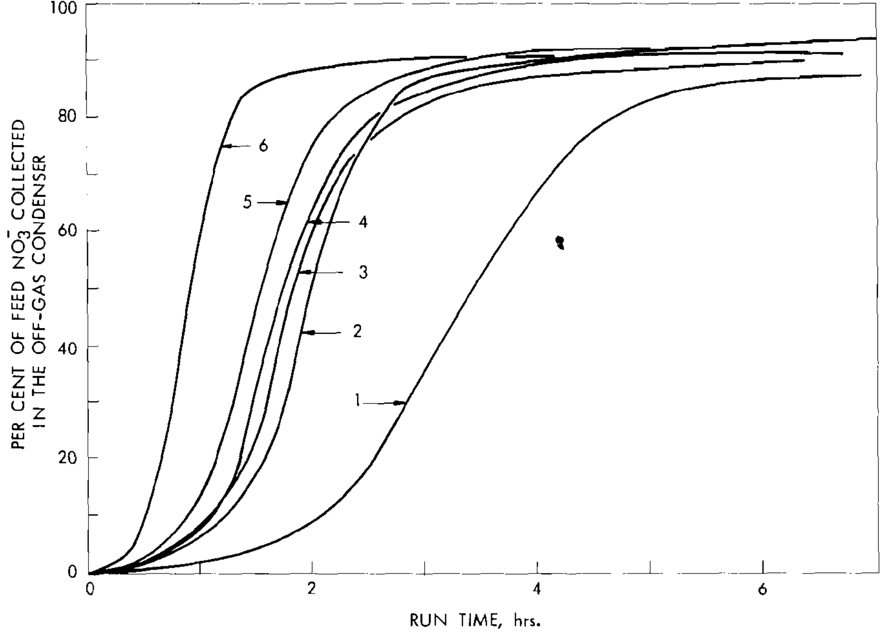

Fig. 4.3. Nitrate collection in the condenser as a function of run time.

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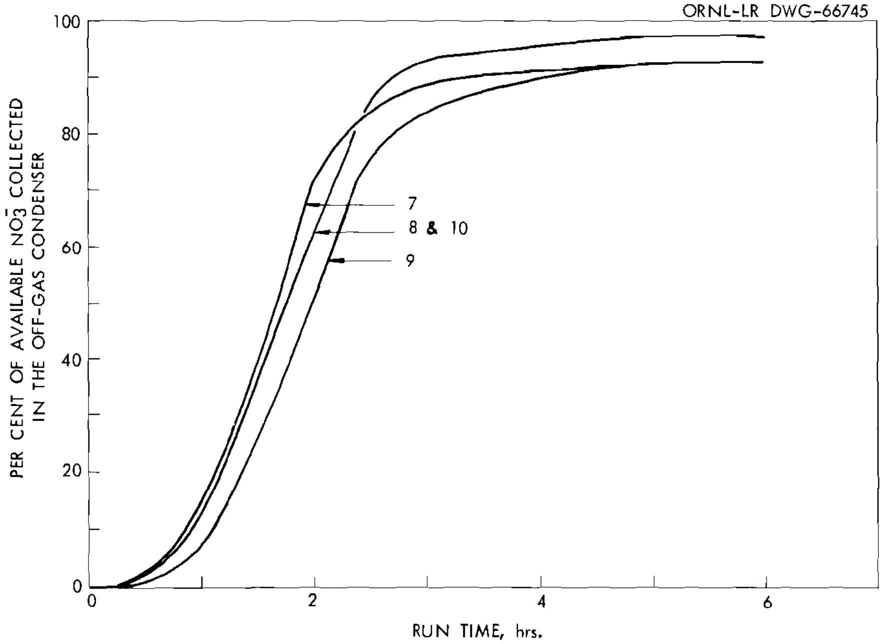

Fig. 4.4. Nitrate collection in the condenser as a function of run time.

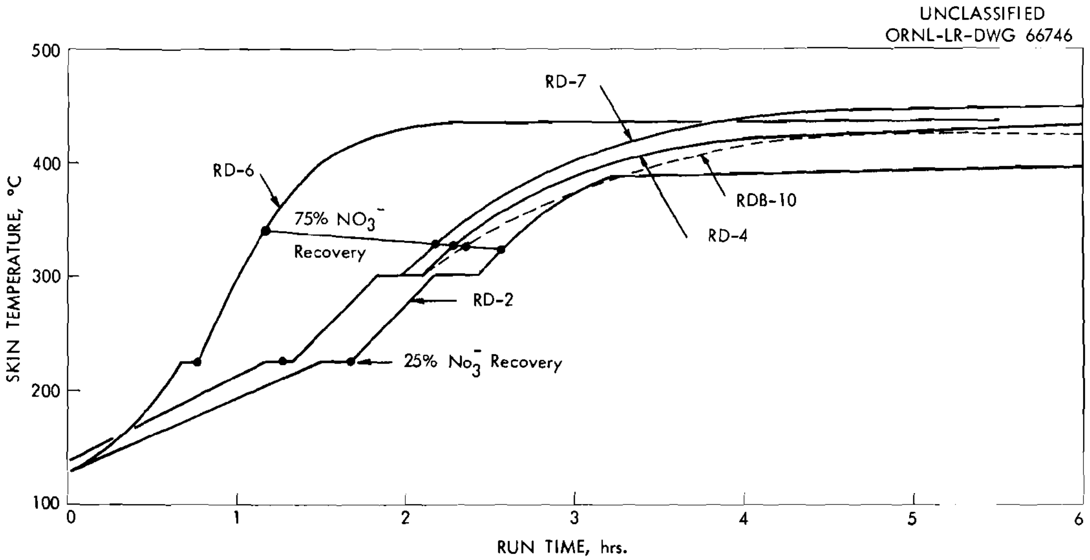

Fig. 4.5. Rotating skin temperature of the rotary denitrator versus run time. Thermocouple located 4 in. from enlarged section near steam inlet end.

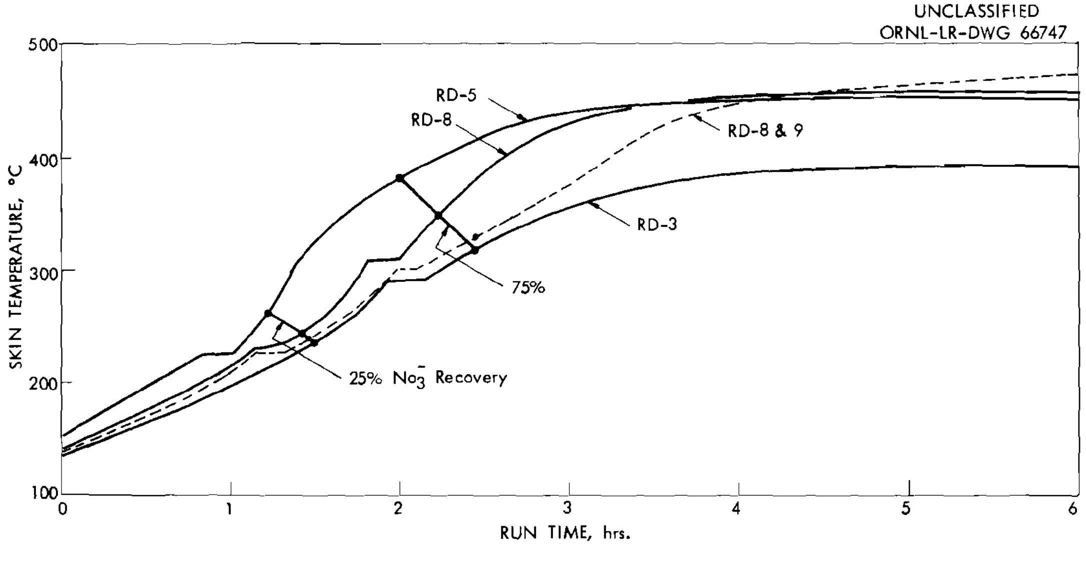

Fig. 4.6. Rotating skin temperature of the rotary denitrator versus run time. Thermocouple located at the drum's center.

The product from RD-2 was dispersable with only ~7% of the creamy fraction. The sols produced from this product were of poor sol quality as judged by previous standards. The gel fragments were lower in density by 2 to 4% over previously acceptable products. The N/Th ratio was 0.078 for this run which is in the range of interest, however, slightly on the high side.

Nineteen kilograms of 5/8-in.-dia 316 stainless steel balls were charged with the thorium nitrate crystals into the denitrator for run RD-3. The creamy fraction was reduced to ~1% for this product, yet its nitrogen content was reduced only 13% from RD-1.

Runs RD-4 through RD-8 were dispersable to > 99%. The amount of creamy fraction, while negligible on a weight basis, caused an easily detectable difference in appearance. These products yielded sols which were milk white even when diluted. This is in contrast to a blue color obtained from a good sol.

All runs from RD-4 through the last RDB-10 were of the proper N/Th ratio to produce good sols. Only the last two, RDB-9 and RDB-10, gave products which had the characteristic appearance of good sols.

The cause of the creamy fraction is unknown and its appearance would not be expected from the analyses. From the analyses, based on previous studies with the agitated trough calciner, one would have predicted that all ten runs would have been acceptable sol producing products.

4.4 Interpretation of Results

The run conditions responsible for the creamy fraction during the first eight runs is not known. The decreasing amount of creamy fraction with successive runs would agree with an initial contamination gradually removed from the system. Runs RD-5 and ATC-55 eliminate both feed and steam as contamination sources. Run ATC-55 was made in the agitated trough calciner using thorium nitrate crystals from the same drum as used for run RD-5. Both runs were made at the same time to check steam contamination. The product from ATC-55 was normal in all respects while that of RD-5 contained the creamy fraction.

The apparent affect of a baffle to increase the steam velocity over the bed during denitration was to cure the creamy fraction. However, the lower steam rate used during run RDB-9 due to a diaphragm rupture in the steam system would tend to minimize the advantage of the baffle. Therefore, the run condition responsible for the creamy fraction is yet undetermined. The effects of steam program variations will be investigated further. The cause of an initial contamination, if it existed, will not be determined unless this effect reoccurs.

5.0 TRANSURANIC STUDIES

A. D. Ryon

The data reported this month is part of the evaluation and improvement studies of the contacting equipment* used in the Transuranics Studies program. The flowsheet consisted of the extraction of rare earths from a 1.5 M $Al(NO_3)_3$ solution by 30% TBP in Amsco.

5.1 Installation of Weir - F. L. Daley

It is evident from the McCabe-Thiele plot of run C-11 (ORNL CF 60-12-28) that extensive backmixing of the organic phase took place, resulting in poor stage efficiency. Careful balancing of the throughput and mixing speed is necessary to reduce the magnitude of backmixing.

The mixer-settler was modified by installing a submerged weir (Figure 5.1, ORNL Dwgs D-45758 and D-45761) between each settler and the downstream mixer and an organic recycle passage between each mixer-settler pair. The purpose is to balance the static head in the settler with an equal head on the downstream side of the weir to maintain the aqueous-organic interface independent of mixer speed. When the mixer speed is greater than that required to pump the aqueous phase, organic is recycled from the settler without loss of the interface, thus eliminating backmixing of the organic from the upstream stage. As the mixer speed is increased further the pressure drop through the organic recycle may become equal to the static head across the weir and the interface will drop.

Hydraulic tests were made in the modified mixer-settler to evaluate the performance of the weir. Aqueous solutions used ranged in specific gravity (25°C) from 1.0053 to 1.2420, viscosity (30°C) from 0.8074 to 5.958 centipoise while the specific gravity of the organic solutions ranged from 0.7648 to 0.9547, viscosity from 0.9241 to 6.046 (Table 5.1). In each test a mixing speed to give a good dispersion was chosen and the maximum throughput without flooding was determined. Flooding was due to dispersion and not in any case caused by inadequate pumping. The aqueous flow was then stopped and the mixer speed required to pump out the aqueous-organic interface determined (Table 5.1). Within the wide range of conditions reported, with the exception of no aqueous flow, the weir was adequate in holding the aqueous-organic interface in the settler thus preventing organic backmixing.

The data in Table 5.1 show that as the specific gravity difference between the aqueous and organic phases is increased, the mixer speed required to make a good dispersion and the mixer speed required to pump out the aqueous-organic interface also increased.

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* A mixer-settler of Belgian design, ORNL drawings D-34200, 34201, 34202, 34203, and 34205.

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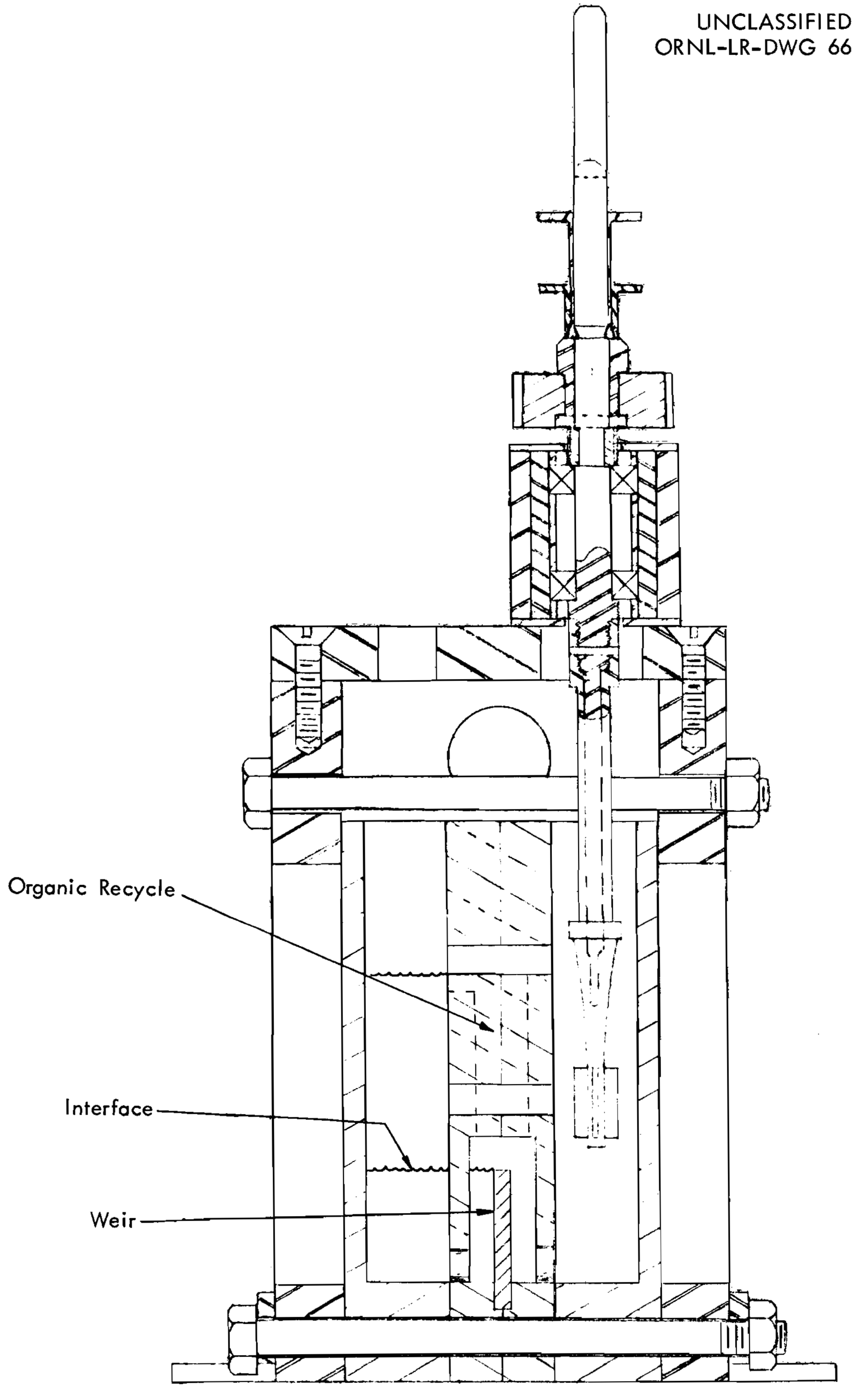

Fig. 5.1. Sectional view of modified mixer-settler.

Table 5.1. Weir Hydraulic Data

Test Solutions	Specific Gravity (25°C)	Specific Gravity Difference Aq-Org	Viscosity Centipoise (30°C)	Mixer Speed Required for Good Dispersion rpm	Maximum* Flow Rate ml/min	Mixer Speed Required to Pump Out Aq-Org Interface at No Aq Flow rpm
1 wt % HNO_3	1.0053		0.807		20	
85% TBP in Amsco	0.9547	0.0506	3.31	1000	20	> 1000
10 wt % HNO_3	1.0335		0.826		20	
85% TBP in Amsco	0.9547	0.0788	3.31	1000	20	> 1000
1.5 M HCl	1.0168		0.872		10	
30% alamine 336 in DEB	0.8713	0.1455	6.05	1300	10	1400
1.5 M HCl	1.0168		0.873		35	
Di Ethyl Benzene	0.8603	0.1565	0.924	1300	35	1700
					10	
				1500	60	1700
1 wt % HNO_3	1.0053		0.807		20	
5% TBP in Amsco	0.7648	0.2405	1.26	1850	30	2150
11 M LiCl	1.2420		5.96		8	
30% alamine 336 in DEB	0.8713	0.3707	6.05	1600	8	> 2000
11 M LiCl	1.2420		5.96		20	
5% TBP in Amsco	0.7648	0.4772	1.26	2770**	30	> 2770

* At mixer speed required to give good dispersion.

** Poor dispersion.

The maximum throughput, at the mixer speed required to give a good dispersion, decreased as the viscosity of the continuous organic phase increased. As shown in Figure 5.2, the throughput is proportional to $(\mu)^{-0.7}$. The viscosity of the dispersed aqueous phase seems to have little effect on mixer-settler throughput.

Comparison of the McCabe-Thiele plots of run C-11, run C-10 (ORNL CF 60-12-28), run MS-29 (Figure 5.3, Table 5.3) and run MS-30 (Figure 5.3, Table 5.3) shows that organic backmixing was eliminated in runs MS-29 and MS-30 even though the operating conditions were much more severe, i.e. high mixer speed and lower flow rate, than those of run C-11 (mixing speed 1800 rpm, total throughput 66 ml/min).

5.2 Effect of Impeller Design

Due to the evidence of poor mixing by the pump impellers (ORNL drawing 34202) in runs C-9 and C-11 (ORNL CF 60-12-28), four blade stainless steel* and four blade Teflon impellers* were tested in the Belgian mixer-settler.

Runs C-9 and C-11, using the pump impeller gave an average stage efficiency for Ce extraction of ~59% at 1800 rpm. In run MS-29 (Table 5.2) the four blade Teflon impeller* was used giving an average stage efficiency of ~93% at 1725 rpm. Run MS-20 (Table 5.4) was made using four blade stainless steel impellers* and average stage efficiency of ~90% at 1400 rpm was realized.

5.3 Effect of Phase Ratio

The phase ratio (aqueous flow/organic flow) at which the Belgium mixer-settler is operated has a marked effect on Ce extraction stage efficiency for the TBP-$Al(NO_3)_3$ flowsheet. As the phase ratio approached unity the stage efficiency of the mixer-settler was increased. Run MS-28 (Table 5.5) operated at a phase ratio (A/O) of 8.4 gave an average efficiency of 45% while run MS-29 (Table 5.2), at a phase ratio of 1, gave an average efficiency of 93%. Since there was no evidence of backmixing in run MS-28 (A/O of 8.4), the low efficiency was probably due to insufficient contact.

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* Four blade impeller, 13/32-in.-dia x 1/2-in. high.

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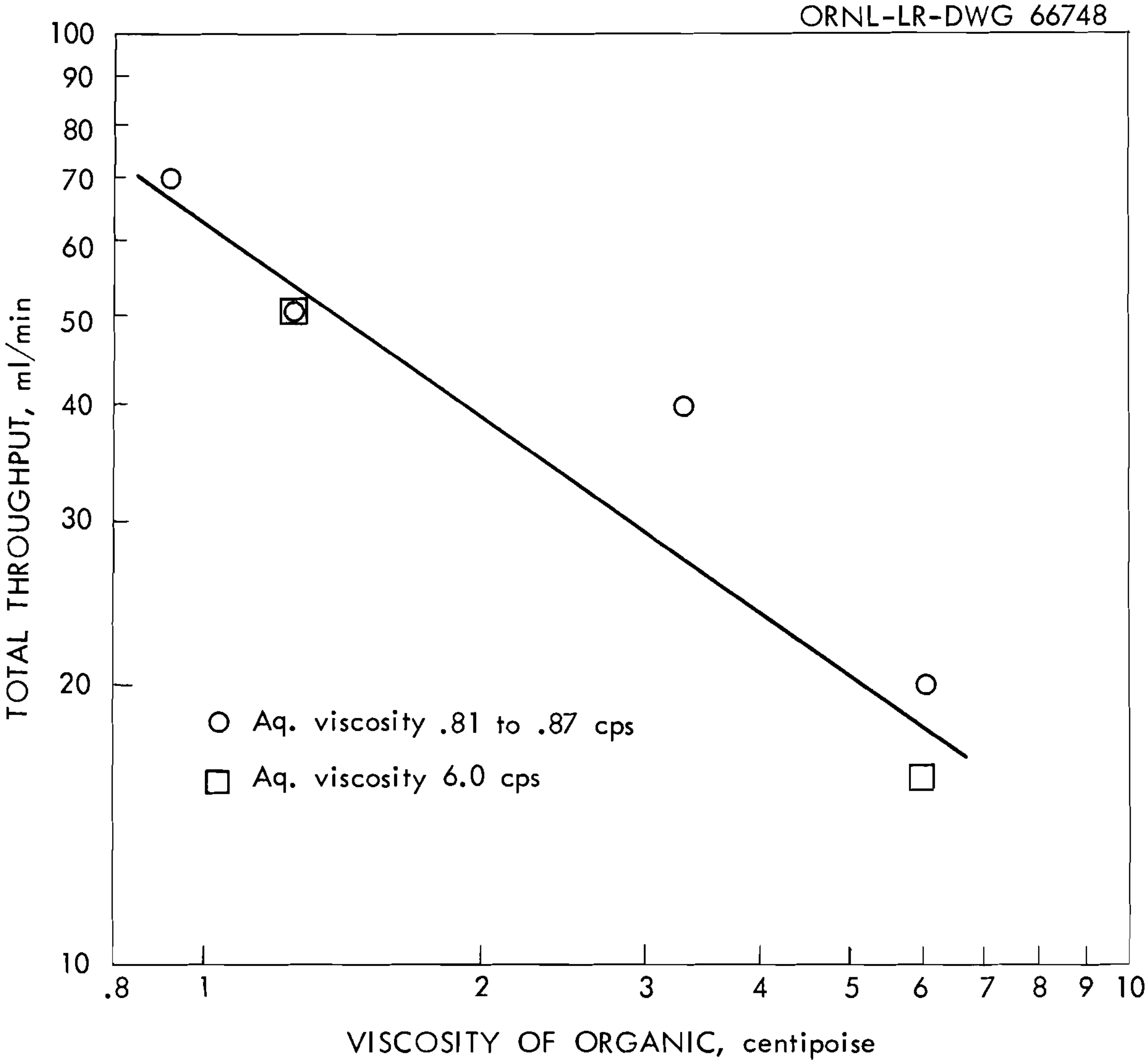

Fig. 5.2. Effect of viscosity on throughput.

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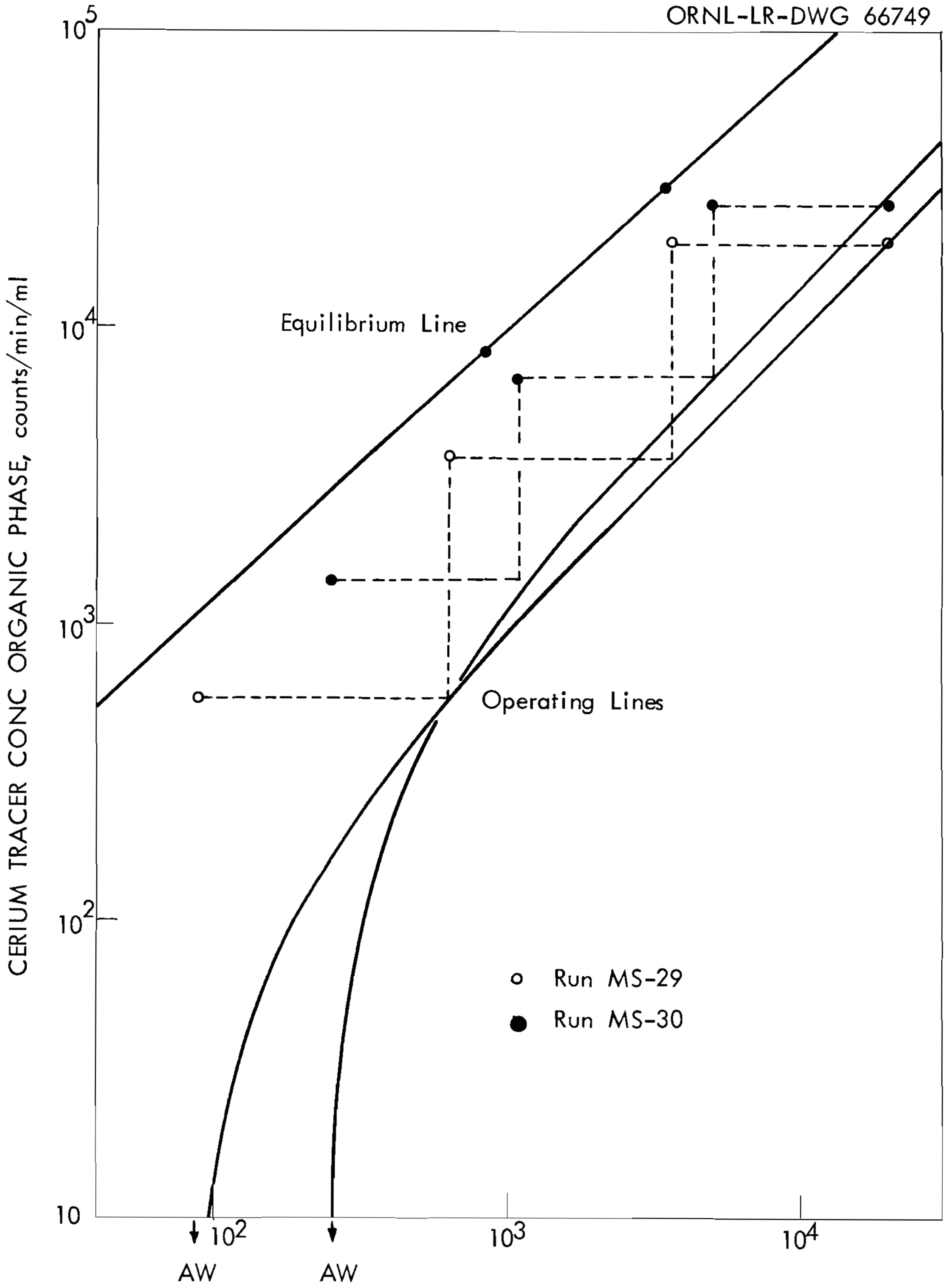

Fig. 5.3. McCabe-Thiele plot for extraction Runs MS-29, MS-30.

Table 5.2. Ce Extraction Run MS-29 Data

Feed:	1.4 M $Al(NO_3)_3$ 0.5 g/liter RE Ce tracer 19,400 γ counts/min/ml	Flow: 20 ml/min
Organic - 30% TBP in Amsco		Flow: 20 ml/min

Impeller - Four blade Teflon

Mixing speed - 1725 rpm

Extraction Stage No.	Ce γ counts/min/ml Flowing Streams Aq	Flowing Streams Org	Equilibrated Samples (MS-30) Aq	Equilibrated Samples (MS-30) Org	Stage* Efficiency, %
1	3,580	19,300	3,460	29,800	92
2	634	3,640	837	8,340	93
3	87	576	-	-	95

* Murphee efficiency based on aqueous phase calculated by use of a McCabe-Thiele plot.

Table 5.3. Ce Extraction Run MS-30 Data

Feed:	1.4 M $Al(NO_3)_3$ 0.5 g/liter RE Ce tracer 19,400 γ counts/min/ml	Flow: 8.1 ml/min
Organic - 30% TBP in Amsco		Flow: 5.5 ml/min

Impeller - Four blade Teflon

Mixing speed - 2160 rpm

Extraction Stage No.	Ce γ counts/min/ml Flowing Stream Aq	Flowing Stream Org	Equilibrated Samples Aq	Equilibrated Samples Org	Stage* Efficiency, %
1	4,860	26,700	3,460	29,800	88
2	1,060	6,880	837	8,340	93
3	250	1,450	-	-	90

* Murphee efficiency based on aqueous phase calculated by use of a McCabe-Thiele plot.

Table 5.4. Ce Extraction Run MS-20 Data

Feed: 1.4 M $Al(NO_3)_3$ Flow: 20 ml/min
0.5 g/liter RE
Ce tracer 23,800 γ counts/min/ml

Organic - 30% TBP in Amsco Flow: 20 ml/min

Impeller - Four blade stainless steel

Mixing speed - 1400 rpm

Extraction Stage No.	Ce γ counts/min/ml Flowing Streams Aq	Org	Equilibrated Sample Aq	Org	Stage* Efficiency, %
Feed Stage	-	-	2,780	40,900	
1	3,140	19,500	-	-	92
2	653	3,250	-	-	86
3	103	564	-	-	90

* Murphee efficiency based on aqueous phase calculated by use of a McCabe-Thiele plot.

Table 5.5. Ce Extraction Run MS-28 Data

Feed: 1.4 M $Al(NO_3)_3$ Flow rate - 44.3 ml/min
0.5 g/liter RE
Ce tracer 19,400 γ counts/min/ml

Organic - 30% TBP in Amsco Flow - 5.3 ml/min

Phase rates A/O = 8.4

Impeller - Four blade Teflon

Mixing speed - 2160 rpm

Extraction Stage No.	Ce counts/min/ml Flowing Stream Aq	Org	Equilibrated Samples Aq	Org	Stage* Efficiency, %
1	18,600	81,100	12,900	72,200	22
2	17,100	75,900	15,400	80,200	35
3	14,100	66,400	13,300	70,700	58
4	9,100	44,700	7,000	46,900	65

* Murphee efficiency based on aqueous phase calculated by use of a McCabe-Thiele plot.

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