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CHEMISTRY-SEPARATION PROCESSES FOR URANIUM PROGRESS REPORT

AEC RESEARCH AND DEVELOPMENT REPORT

LABORATORY RECORDS

TECHNICAL DIVISION

M.D.Peterson, Director

CHEMICAL TECHNOLOGY

PROGRESS REPORT FOR QUARTER ENDING AUGUST 31,1949

F.L.Steahly, Associate Director

EDITED BY

W.K.EISTER

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TECHNICAL DIVISION

M. D. Peterson, Director

CHEMICAL TECHNOLOGY
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F. L. Steahly, Associate Director

edited by
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DATE ISSUED OCT 20 1949

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ABSTRACT

The chemical development progress of the Laboratory, Semi-Works, Pilot Plant and Design Sections of the ORNL Technical Division for the quarter ending September 31, 1949 is submitted.

The problems investigated included UAP and TBP process for uranium recovery from metal waste; Redox, "23", "25", RaLa, and dry fluoride processes; and ORNL radiochemical waste disposal.

SUMMARY

Redox pilot plant runs at full Hanford radioactivity level demonstrated adequate uranium decontamination from fission products ($> 3 \times 10^5$) in two cycles under ORNL acid deficient conditions. The uranium loss through two cycles was 0.01 to 0.02%. The increase in first cycle extraction plutonium loss (0.1 to 1.0%) when going to full Hanford activity level was apparently due to the reduction of plutonium VI. The cause of this reduction has not been determined definitely.

The semi-works demonstration of the UAP process for uranium recovery from metal waste was completed. The process gave a uranium loss of 0.5 to 1.0% with a gross fission product decontamination factor of 10^3.

Studies with the tributyl phosphate solvent extraction process for waste uranium recovery demonstrated that plutonium IV was extractable under the proposed process conditions and that "Varsol" may be used instead of hexane as a diluent for tributyl phosphate. This solvent mixture has satisfactory physical properties, including flash point greater than that for hexone (105° F). Other work indicated that feed solution as dilute as 0.1 molar uranium may be processed with satisfactory yields and decontamination. The flooding rates for the 1A and 1B column were 1000 and 500 gallons per hour per square foot, respectively, using 1/4 × 3/8 inch split rings in a 1½ inch diameter packed column. The HETS was 2.5 feet at 25% of flooding in a 1½ inch diameter column packed with 1/4 × 3/8 inch split rings.

An extraction process using 50% tributyl phosphate and 50% hexane as solvent for the recovery and decontamination of thorium from 23 raffinates has been developed. Countercurrent batch extraction runs indicate that thorium

recovery of greater than 99.9% and beta decontamination factors of 1700 are obtainable.

The radiochemical waste evaporator has been in operation approximately 90 days at feed rates of 300 to 600 gallons per hour obtaining a condensate decontamination factor of 10^3 and a volume reduction of about 4 to 50 depending upon the type of waste being evaporated.

In the RaLa process development, both filtration and centrifugation were successfully applied to the sulfate precipitation and metathesis steps and the barium losses were significantly reduced. Filtration was selected for further development.

The ion exchange barium purification has been successfully demonstrated on full chemical scale.

Pilot plant runs demonstrated that satisfactory yield and decontamination were obtained in two cycles with no ferrous sulfamate in the first cycle. Ferrous sulfamate was omitted because of the high mercuric ion concentration in the metal solution.

REDOX PROCESS STUDIES

INVESTIGATION OF PILOT PLANT PLUTONIUM LOSSES

Considerable laboratory work has been carried out in an effort to determine causes for the losses experienced in the final pilot plant runs made at full Hanford radiation level. Losses increased from less than 0.1% at the beginning of these runs, to the order of 1%. This 1% loss persisted throughout, despite efforts to maintain strict control of the process conditions. Although larger than usual amounts of americium and curium were determined to be present in these feeds, it was decided beyond reasonable doubt that the increased losses, of the order indicated above, were plutonium losses.

By determinations of plutonium extractibility in process control samples it was generally observed that the oxidized feeds, prior to filtration and transfer to the columns, contained 99.9+% plutonium extractable with basic flowsheet conditions, yet actual column losses were observed to be consistently in the order of 0.5 to 1.0%. Analyses of the 1AW samples showed that the plutonium lost in the process was definitely in an inextractable state under basic conditions; however, it was also shown that essentially 100% of this plutonium was extractable with hexone containing 0.5 *N* HNO_3. This latter observation led to the conclusion that a stable plutonium polymer was not responsible for the losses encountered.

Investigations by batch extraction were made in an attempt to point out unusual conditions which might have affected plutonium losses (Table 1). Those conditions which were considered as likely possibilities included occlusion on suspensoids of insolubles introduced by poor filtration or crud formation and, possibly, reduction by hexone impurities. The latter seemed to be ruled out since negative results were obtained in batch tests with the currently used hexone which was known to have a reducing normality of less than 0.002 *N*. Too, no losses were observed during lengthy hexone-aqueous contact in simulated extraction or scrubbing sections.

The effect of suspensoids remained as the most probable cause. Quantitative evaluation was improbable due to the complexity of the problem involved, but it was known that considerable likelihood existed for introduction of such insolubles. A faulty filter and unclean displacement fluid were primary suspects.

TABLE 1

Effect of Various Treatments on the Amount of Inextractable Plutonium in Oxidized ORNL Redox Feeds

REAGENT OR EFFECT	CONDITIONS STUDIED	PERCENT Pu LOSS	
		Control	After Treatment
Local Reduction	0-0.01 *M* $FeSO_4$ (ppt. formation)	0.04	30.0
Periodate	0-0.01 *M* H_5IO_6 (24 hr. contact)	0.4	6.0
Persulfate	0-0.01 *M* $(NH_4)_2S_2O_8$ (24 hr. contact)	0.4	5.0
Hypochlorite	0-01% NaOCl (24 hr. contact)	0.4	4.0
Adipate	0-0.02 *M* Adipic Acid (exceeding solubility)	0.04	2.1
Ageing	1 day - 6 weeks standing (stored samples)	0.04	0.78
Hexone Impurities	<.002 *N* - .008 *N* reducing power (commercial)	0.15	0.45
Ceric	0-0.01 *M* Ce^{+4} (ppt. formation)	0.4	1.0
Displacement Fluid	0-2%/vol methyl cycle hexane (30 min contact)	No change	
Cleaning Agent	0-0.01 *M* NH_4HF_2	No change	
Organic Acids	0-2%/vol butyric and/or valeric acids	No change	
Ferric	0-0.01 *M* $Fe(NO_3)_3$	No change	
Sulfate	0-0.02 *M* Na_2SO_4	No change	
Radiation Level	25-100% Hanford level	No change	
Column Reduction	Simulated Extraction and/or Scrubbing Sections	No change	

However, it was possible to obtain only one laboratory sample of the feed after filtration and prior to displacement to the column. Run 50R was stopped seven hours before completion; the remaining feed was forced back into the displacement tank A-12 for sampling, and this solution was found to have 5.0% inextractable plutonium. A sample of the process displacement fluid, methyl-cyclohexane, was then found to contain a suspended white substance which was observed to be contaminated with plutonium. Later washing of the MCH catch tank revealed that a large amount of a white, insoluble organic compound of uranium had collected, a considerable portion of which was noted to distribute readily in the organic phase. It was thus felt that MCH stability was questionable, and it seemed possible that resulting decomposition products could add either suspensoids or reducing agents to plant streams. Inclusion of adipic acid, a possible decomposition product, in feeds was found to cause increased losses, although adipic acid has not been positively identified as the organic radical of the MCH crud. A precipitate formed by the addition of 0.01 *M* ferric sulfate to a feed sample was observed to cause an abnormally high loss, whereas addition of lower quantities, insufficient to cause a permanent precipitate, had no effect on losses. The need for a general clean-up of plant equipment, as well as filter repair has been demonstrated as necessary to remove unwanted solids from the system.

PLUTONIUM RECOVERY FROM PILOT PLANT IBP SOLUTIONS

The pilot plant has in storage (Tank E-2, 1100 gallons, and Tank F-2, 350 gallons) IBP solution from the Redox experimental and test runs. A process for plutonium recovery and decontamination was desired before returning this product to Hanford (Table 2). Studies have been made to determine whether the present second cycle plutonium flowsheet conditions were satisfactory or if significant changes were necessary.

TABLE 2

Specifications for Plutonium Returned to Hanford

COMPONENT	SPECIFICATION
Plutonium	10 gms Pu/liter
Iron	< 5.0% (of total plutonium)
Aluminum	< 0.5% (of total plutonium)
Chromium	< 0.2% (of total plutonium)
Uranium	< 40-50 mg/gm Pu
Gamma Activity	< 10^7 d/m/gm Pu
Beta Activity	< 10^8 d/m/gm Pu
Hexone	None

TABLE 3

Analysis of 1BP Solutions

COMPONENT	TANK E-2	TANK F-2
Specific Gravity	1.178	1.160
Aluminum Nitrate	ca 1.2 *M*	ca 1.0 *M*
Reducing Molarity	0.03 *M**	
Oxidizing Molarity		0.08 *M* ($Cr_2O_7^{=}$)
Nitric Acid	0.13 *M*	0.23 *M*
Plutonium	3.9×10^8 α/m/ml	6.4×10^8 α/m/ml
Total β	2.2×10^7 c/m/ml	7.0×10^5 c/m/ml
Total γ	4.3×10^4 c/m/ml	2.5×10^3 c/m/ml
Ruthenium β	1.5×10^6 c/m/ml	2.9×10^5 c/m/ml
Zirconium β	1.1×10^6 c/m/ml	
Cerium β	1.3×10^7 c/m/ml	

* This will be adjusted to ca 0.1 M oxidizing.

The solution from Tank E-2, containing the plutonium from the earlier experimental runs had been expected to present considerable difficulties, especially in decontamination. However, it has been determined that a β decontamination factor of 10^4 is possible in a single cycle and it has been estimated that specifications will be easily met in a second cycle (Table 5). Plutonium losses of less than 0.01% have also indicated that product yields will also be satisfactory (Table 4).

The solution from F-2, containing the plutonium from the latter full Hanford level test runs (Table 3), was considered normal as a feed for the second cycle plutonium process, and it appears that a single cycle allows a β decontamination factor of only 10^1 to 10^2 with a plutonium loss of only 0.01%. However, since the decontamination was not adequate, a second cycle will definitely have to be investigated to determine if specifications for decontamination can be met.

It is planned to extend this study through a two-cycle process, utilizing experiments with the laboratory countercurrent extractor.

PILOT PLANT DEVELOPMENT

The two previous Technical Division Quarterly reports, ORNL 268 and 323, presented the Pilot Plant results of the testing of the acid ANL Redox Flowsheet and the development of the acid deficient ORNL Redox Flowsheet. Comparison of the two flowsheets showed that the acid deficient flowsheet was superior, in that decontamination was increased an order of magnitude without appreciably affecting either uranium or plutonium losses. The objective of the Redox development program in the Pilot Plant in the past period was to make six runs at full Hanford concentration of both plutonium and fission products to verify the previous results obtained under acid deficient conditions. This work completed the ORNL Redox program.

The ORNL-ORNL #1 acid deficient flowsheet combination was used in these runs and resulted in two-cycle uranium decontaminations of 5-10 $\times 10^5$ for beta, and 3-8 $\times 10^5$ for gamma activity. These results slightly exceeded the process specification for gamma decontamination of 3 $\times 10^5$. This degree of decontamination yielded a uranium product which had a beta and gamma activity of 20% and 200%, respectively, of the equilibrium activity of natural uranium. The plutonium decontamination obtained for the first cycle was 2 $\times 10^4$ for beta

TABLE 4

Plutonium Losses Observed in Exhaustive Batch Extraction of 1BP Solutions

FEED:	E-2 3.9×10^6 α/m/ml	F-2 6.4×10^6 α/m/ml
CONDITIONS:	1.2 M $Al(NO_3)_3$	1.0 M $Al(NO_3)_3$
	N. HNO_3 as indicated	N. HNO_3 as indicated
	0.1 M $Na_2Cr_2O_7$	0.08 M $Na_2Cr_2O_7$

ORGANIC: Hexone, containing HNO_3 as indicated.

METHOD: Twelve consecutive equal volume passes of hexone with a single feed. Plutonium alpha determined by LaF_3 and TTA in the resulting raffinates.

N. HNO_3 IN SYSTEM	PERCENT LOSS (TANK E-2)		PERCENT LOSS (TANK F-2)	
	LaF_3	TTA	LaF_3	TTA
+ 0.3 *N*	0.03	0.003	0.02	0.0008
+ 0.0 *N*	Precipitate Formed		0.048	0.031
0.1 *N*	*Precipitate Formed*		*Precipitate Formed*	

TABLE 5

Plutonium Losses and Decontamination Observed in Counter-Current Batch Extraction

CONDITIONS:	E-2 a	E-2 b	F-2 a	F-2 b
FEED:	1.2 M $Al(NO_3)_3$	1.2 M $Al(NO_3)_3$	1.0 M $Al(NO_3)_3$	1.0 M $Al(NO_3)_3$
	0.1 M $Na_2Cr_2O_7$	0.1 M $Na_2Cr_2O_7$	0.08 M $Na_2Cr_2O_7$	0.08 M $Na_2Cr_2O_7$
	0.13 N HNO_3	0.3 N HNO_3	0.23 N HNO_3	0.23 N HNO_3
SCRUB:	1.3 M $Al(NO_3)_3$	1.3 M $Al(NO_3)_3$	1.3 M $Al(NO_3)_3$	1.3 M $Al(NO_3)_3$
	0.01 M $Na_2Cr_2O_7$	0.01 M $Na_2Cr_2O_7$	0.01 M $Na_2Cr_2O_7$	0.3 N HNO_3
	0.3 N HNO_3	0.3 N HNO_3	0.3 N HNO_3	0.3 N HNO_3
ORGANIC:	Hexone, 0.0 N HNO_3	Hexone, 0.3 N HNO_3	Hexone, 0.0 N HNO_3	Hexone, 0.3 N HNO_3
FLOW RATIO:	F/S/O = 1/1/2	Stages: 8 Ext'n, 6 Scrub		Volume Changes: 2.5

OBSERVED VALUES	E-2 a	E-2 b	F-2 a	F-2 b
Pu D. C. (O/A)	0.7	2.1	3.0	8.7
Pu Loss (7 stages)	64.0%	0.04%	0.06%	0.02%
β D. F. (Extraction)	0.8×10^3	2.7×10^3	14	8
β D. F. (Overall)	2.0×10^4	1.1×10^4	42	23
Ru β D. F. (Overall)	3.6×10^3	3.5×10^3	21	12
γ D. F. (Overall)	0.8×10^3	1.0×10^4	ca 100	ca 100

and 8×10^3 for gamma. The results of these high activity level runs were consistent for both gross and individual fission product contamination, and were consistent with previous data from lower activity level runs. The total uranium losses for two cycles was found to be less than 0.01%, and the plutonium losses in these high level runs averaged approximately 1%. From the data obtained it was concluded that the ORNL Flowsheet met the uranium decontamination requirements for the Redox process. Additional experimental work will be required to determine the cause of the increased plutonium loss.

First Cycle Feed Preparation. The material processed in the six 100% Hanford activity level runs was 8 in. Hanford production slugs having an average exposure time of 700 days in the Hanford piles. This exceptionally long irradiation history was very conductive to transplutonic element formation, and the interpretation of the plutonium loss data was complicated by the presence of inextractable americium and curium alpha activity.

The slug jackets were removed by the usual $NaOH$-$NaNO_3$ method, and the uranium was dissolved in nitric acid. The acidity of the final dissolver solution for the six runs was varied from 0.4 *N* acid to 0.3 *N* acid deficient in order to study the effect of this variable on plutonium losses.

The oxidation of the feed was carried out by making the solution 0.1 *M* in sodium dichromate and heating at 85° C for six hours. Acid adjustments were made by the direct addition of concentrated sodium hydroxide.

Solution clarification was accomplished by vacuum filtration of the feed solution through sintered stainless steel. The stainless steel filter element was known to have been previously fractured, permitting small particles to pass through. However, this incomplete filtration did not affect the column operations in any manner.

Overloading of the dissolver caused plugging of the slug chute. The plug was removed by filling the dissolver and lower part of the chute containing the slug with HNO_3. The acid was then heated and the slugs dissolved.

First Cycle Column Operation. The data obtained in the high level first cycle runs with the acid deficient flowsheet verified all information obtained on low level processing with the exception of plutonium losses (Table 6). The decontamination, uranium losses, and mechanical operability of the columns were normal and no difficulties were encountered. The mechanical operability of the columns have been without incident and no column fouling of any sort have been evidenced during 3000 hours of column operation. This lack of column flow interruption has been evidenced in spite of the fact that appreciable solid particles have entered through both the feed and scrub streams.

TABLE 6

ORNL Pilot Plant Data—
Redox First Cycle Column Data Summary

ORNL Flowsheet Conditions Activity
Level 100% Hanford

RUN	TOTAL LOSSES (PERCENT)		PLUTONIUM DECONTAMINATION x 10^{-3}					URANIUM DECONTAMINATION x 10^{-3}				
	Pu	U	Beta	Gamma	Ceβ	Ruβ	Zrβ	Beta	Gamma	Ceβ	Ruβ	Zrβ
48	0.7	.005	27	12	3000	1.9	11	19	12	20,000	0.9	21
49	1.9	.005	20	8	3300	2.9	5	8	4	60,000	0.5	20
50	1.5	.002	20	6	3500	2.2	7	6	3	30,000	0.4	10
51	.5	.003	23	8.5	4700	2.1	9	6	5	7000	0.4	19
52	.2	.002	33	13	170	2.0	8	14	8	1000	0.8	32
53	.7	.01	19	5	2300	1.6	6	7	235	1400	0.5	8

The uranium losses obtained less than 0.01%, show more than adequate recovery of uranium, the losses being equally divided between the extraction and stripping raffinates. The decontamination values obtained were highly consistent and in line with previous runs made at 30% Hanford activity. Ruthenium was the most consistently behaved fission product, followed by cerium, zirconium and columbium in that order. The gamma activity in the plutonium (IBP) stream was split evenly between zirconium, columbium, and cerium. In the uranium product (ICU) stream ruthenium accounted for 60-90% of the gamma activity, the remaining being due to zirconium and columbium.

The results of laboratory extractions performed on feed and aqueous waste samples indicated that the bulk of the plutonium losses were due to incomplete oxidation of the feed. The alpha activity in the aqueous waste stream was not extractable under acid deficient conditions, but was easily extracted under acid conditions. This indicated that the plutonium in that stream was not polymerized, and that it was present in the plus four state. There was some evidence that a small part of the losses were caused by reduction of plutonium after the feed preparation step.

During the latter portion of one run the hexone extract was made 0.2 *N* in nitric acid. Because of the low plutonium loss (0.1%) on this particular run, no firm conclusion could be made on the effect of the acidic hexone on plutonium losses, although decontamination decreased by a factor of 100.

Second Cycle Feed Preparation. The feed for the second uranium cycle was prepared by concentration of the aqueous uranium stream from the first cycle to 2 *M* and adjusting to 0.2 *N* acid deficiency with NaOH. No operating difficulties were encountered except that the material for Run 50 was lost due to gasket failure in the drain line of the second cycle feed make-up tank.

Second Cycle Column Operation. The data from the second uranium runs showed that the acid deficient ORNL Flowsheet conditions were adequate to meet Hanford process specifications for uranium decontamination (Table 7). The uranium losses in this cycle averaged less than 0.01%. The decontamination factors varied from 50 to 170, this range of values being attributed primarily to variations in decontamination obtained in the previous cycle.

The hexone line which carried uranium from the second cycle extraction column to the stripping column plugged in a number of the 100% level runs. This difficulty was subsequently found to have been caused by a water soluble crud composed of oxidized iron, the source of which has not been definitely determined.

TABLE 7

ORNL Pilot Plant Redox Data—
Second Uranium Cycle Column Data Summary

ORNL Flowsheet Conditions First Cycle
Feed Activity 100% Hanford Level

RUN	URANIUM LOSSES (Percent)	DECONTAMINATION FACTOR			
		Beta	Gamma	Ruβ	Zrβ
48	0.013	50	46	40	40
49	0.002	100	110	70	470
50	Material Not Processed in Second Cycle				
51	0.002	85	55	70	45
52	0.008	135	120	120	40
53	0.02	130	170	125	300

TABLE 8

ORNL Pilot Plant Redox Data—
Overall Uranium Decontamination & Losses for Two Cycles

ORNL Flowsheet in First Cycle
ORNL Flowsheet in Second Cycle
Initial Activity 100% Hanford Level

RUN	DECONTAMINATION x 10^{-5}					URANIUM LOSSES (Percent)
	Beta	Gamma	Ru	Zr	Cb	
48	5	4	0.3	5	40	0.02
49	7	6	0.6	10	10	0.01
50	Material Not Processed in Second Cycle					
51	6	3	0.5	10	3	0.01
52	10	8	0.6	5	10	0.01
53	7	5	0.6	20	9	0.02

Uranium Decontamination and Losses for Two Cycles. The data (Table 8) showed the losses for both cycles averaged less than 0.05%, with a gross gamma decontamination of 3-8 × 10^5 and a beta decontamination of 5-10 × 10^5.

A summary of the two cycle fission product decontamination and losses for uranium show that both uranium losses and fission product decontamination specifications were exceeded (Table 8). The results of these 100% Hanford fission product level runs were consistent for both gross and individual fission product decontamination and were also consistent with previous ORNL Flowsheet data at the lower levels of activity. These data further substantiate the consistent reproducible decontamination values and low uranium losses obtained for acid deficient systems in general.

Hexone Recovery. The hexone used in the first four full level runs was recycled material that was water washed and steam distilled from a 3% NaOH heel. Plutonium losses were not affected by giving the recycled hexone a sodium dichromate-nitric acid oxidation before distillation on the last two Hanford level runs.

Analytical Developments. A titration method using ferroin indicator was used for the routine determination of methyl isobutyl carbinol in Pilot Plant hexone. The method was specifically designed for use with hexone containing methyl isobutyl carbinol in the 0.01-0.05% concentration range, and the method is believed to be accurate to ± 0.01% methyl isobutyl carbinol.

Chromium and iron in the extraction raffinates (1AW and 1DW) and aqueous plutonium (IBP) streams interfered in the fluorimetric determination of uranium when the concentration of uranium was below about 1 mg/ml in the IAW stream and below about 0.1 mg/ml in the IBP and IDW streams. A "spiking" technique in which the amount of quenching due to iron and chromium was determined has been used until recently, but the results were somewhat erratic. A preliminary extraction of uranium from chromium and iron is now being made with tri-n-butylamine in hexone in order to eliminate the quenching of the uranium fluorescence due to these elements.

The determination of gamma activity in pilot plant samples was done almost wholly by use of the ion chamber. Gamma analyses by a G-M counter was done on a few feed samples in order that a basis for comparison of the two methods may be maintained. The determination of gamma activity in all samples by both methods was not justified, and the use of the ion chamber was preferred over counting because of the greater convenience and accuracy, especially for low activity samples.

Program. Completion of the six Redox process verification runs at 100% Hanford fission product activity marked the termination of participation of the ORNL pilot plant in Redox development work. It is contemplated that future work on this process by the pilot plant group will be limited to the preparation of the final report, and to the recovery of the plutonium in the stored IBP solutions from pilot plant processing. The plutonium recovery operation will be preformed in the latter quarter of 1949 after conclusion of the 25 process verifications runs. Plutonium recovery will consist of processing the IBP solution through two cycles of solvent extraction, and concentrating the resulting product.

METAL RECOVERY—UAP PROCESS

The development of the Uranyl Ammonium Phosphate Process for the recovery and decontamination of uranium from Hanford metal waste has been completed on a semi-works scale (150-190 liters) in cooperation with K-25 personnel who carried out the initial laboratory development. The process consists of two precipitation-filtration cycles with a precipitation-centrifugation recycle step for recovery of the uranium from the filtrates.

The feed materials for supernatant runs were obtained from the Hanford 103 T and 103 U tanks. For total waste type runs, the supernatants from the Hanford 103 T and 103 U tanks were fortified with UO_2^{++}, $NaPO_4$, $SO_4^{=}$, and NO_3^{-} ions to simulate, ionically, a total waste. However, the slurry contained only 40% of the total waste activity. It was recognized that the fission product spectrum in the simulated total waste runs was not truly representative of the activity in the actual total waste (Table 9).

The average gross β and γ fission product decontamination factors were 1300 and 830 for a series of three simulated Hanford 103 T total waste type runs. This decontamination was somewhat higher than the factors of 470 and 270 for beta and gamma activities, respectively, obtained from a series of six simulated Hanford 103 U total waste runs. Generally, the data were quite consistent and reproducible, except for one run (number 22) which was invalid because an insufficient amount of phosphate ion was added for complete precipitation.

Recycle material from the previous run was added to each run except the first 103 T run and the last three 103 U simulated total waste type runs. The older feed had a definitely higher decontamination factor, but recycling had no noticeable effect upon the decontamination factor over a series of seven runs.

The β decontamination factors for one 103 T and one 103 U supernatant run were 2020 and 727, respectively. The γ factors for the same runs were 1,106 and 294, respectively. These decontamination factors were about 50% higher than for the total waste runs; however, the β activity in the final product was somewhat higher (a factor of 3.8 times the equilibrium activity of natural uranium for 103 T, and a factor of 7.62 for 103 U supernatant runs, versus factors of 2.83 and 5.53 for the corresponding total waste type run.)

The uranium loss was consistently less than one percent, averaging about 0.60%.

All process steps were found to be operable. The first cycle filtration constant was about 40% higher than desired for Hanford operations, although more filtration area could be provided to reduce the time cycle. Second cycle filtration rates were adequate for Hanford requirements.

TABLE 9

Uranyl Ammonium Phosphate Process—Semi-Works Runs

Summary of Uranium Loss and Decontamination Data

PROCEDURE: Two uranyl ammonium phosphate precipitations by the addition of ammonium nitrate, precipitates filtered and the uranium in the filtrates recovered by reprecipitation with sodium hydroxide.

SUPERNATE RUNS: Feed volume 190-200 liters last four of five filtration washes discarded.

SIMULATED TOTAL WASTE RUNS: Feed volume 150-160 liters last three of five filtration washes discarded.

FEED COMPOSITION:	SUPERNATE	TOTAL WASTE
U (*M*)	0.111	0.264
Na(*M*)	3.31	3.91
PO_4(*M*)	0.267	0.330
SO_4(*M*)	0.225	0.276
NO_3(*M*)	0.603	0.710
CO_3(*M*)	0.912	1.09
Gross β (c/m/ml at 10% Geo.)	$1\times34\times10^7$	1.28×10^7
Gross γ (c/m/ml at 10% Geo.)	2.9×10^4	2.6×10^4

TYPE OF FEED MATERIAL PROCESSED	RUN NUMBER	U TOTAL IN FEED -GRAMS	RECYCLE MATERIAL ADDED	FIRST CYCLE U Recycled (Percent)	FIRST CYCLE U Lost in Cycle** (Percent)	FIRST CYCLE Gross β DF	FIRST CYCLE Gross γ DF	SECOND CYCLE U Recycled (Percent)	SECOND CYCLE U Lost in Cycle** (Percent)	SECOND CYCLE Gross β DF	SECOND CYCLE Gross γ DF	RECYCLE U Loss in Effluent (Percent)	OVERALL U Loss (Percent)	OVERALL U Recycled (Percent)	OVERALL Gross β D.F.	OVERALL Gross γ D.F.	RATIO OF β ACTIVITY IN PRODUCT TO β ACTIVITY OF NATURAL U 70 cts/min/mg
Synthetic 103T total waste	Ave. 13-18	10,621	Yes	7.14	0.12	---	---	5.2	0.16	---	---	0.32	0.60	12.34	---	---	
Ionically enriched	19	9,725	No	7.28	0.04	79	41	2.3	0.10	17.7	19.8	0.99	1.13	9.6	1400	811	2.29
103T Supernatant	20	10,750	Yes	8.13	0.06	83	54	3.2	0.04	16.9	13.6	0.33	0.43	11.3	1400	734	3.07
to simulate	21	10,954	Yes	7.77	0.07	68	46	5.83	0.12	17.7	20.5	0.17	0.36	13.6	1200	952	3.13
total waste	Ave. 19-21	10,476		7.73	0.056	77	47	3.78	0.086	17.4	18.0	0.50	0.64	11.5	1330	832	2.83
Ionically enriched	22*	11,033	Yes	16.83	0.18	71	45	7.16	0.22	5.3	3.7	0.34	0.74	24.0	380	168	8.50
103 U Supernatant	23	10,859	Yes	5.53	0.03	58	41	4.68	0.13	7.6	6.0	0.24	0.40	10.2	440	246	6.06
To Simulate	24	11,298	Yes	10.50	0.03	55	41	4.5	0.22	8.7	7.6	0.23	0.48	15.0	520	312	5.33
total waste	25	11,214	Yes	7.24	0.02	67	53	8.0	0.10	9.1	5.8	0.10	0.22	15.2	610	307	5.07
	26	9,778	No	9.24	0.09	56	47	5.3	0.31	12.0	7.8	0.36	0.76	14.5	676	368	6.47
	27	9,600	No	7.77	0.06	92	49	8.7	0.24	7.1	4.3	0.25	0.55	16.5	657	211	4.51
	28	9,743	No	4.89	0.09		16	9.3	0.17		12.2	0.27	0.53	14.2	525	241	5.74
	Ave. 23-28*	10,408		7.53	0.05	65.6	41.2	6.6	0.195	8.9	7.3	0.24	0.49	14.2	568	281	5.53
(U Averages only)	Ave. 19-28*	10,426		7.60	0.05			5.8	0.16			0.33	0.54	13.4			
T03U Supernatant	29	6,578	No	10.6	0.19	55.6	38.2	5.2	0.43	13.1	6.4	0.31	0.93	15.8	727	294	7.62
103T Supernatant	30	5,205	No	11.5	0.14	171	97	4.0	0.40	11.8	11.4	--	--	15.5	2020	1106	3.8

*Results from Run 22 are excluded from averages because feed was deficient in phosphate ion.

**Cycle loss contained in filtration washings.

METAL RECOVERY—TBP PROCESS DEVELOPMENT

CHEMICAL STUDIES

Plutonium and Fission Product Studies. A study of the variables affecting the solvent extraction of plutonium and fission products by tributyl phosphate has been initiated. Under the conditions of the tributyl phosphate process for waste uranium recovery (3.0 *M* HNO_3, 1-3 *M* $NaNO_3$ 0.1 *M* uranium present) the distribution coefficients for cerium III, zirconium, and plutonium IV were approximately 0.001, 0.3, and 1.0, respectively.

Using tracer concentrations of cerium III, plutonium, and zirconium, the effect of nitric acid and sodium nitrate concentrations on the solvent extraction of these elements by 15% tributyl phosphate in hexane was studied in batch extraction. The extraction of cerium III was negligible (distribution coefficient organic/aqueous less than 0.001) in the acid range of 0.2-10 *N* and was not appreciably affected by varying the $NaNO_3$ concentration from 0-3 *N*. The distribution coefficient for zirconium (organic/aqueous) increased from 0.004 at an acid concentration of 0.2 *N* to 0.72 at 10.2 *N*. The distribution coefficient for plutonium IV (organic/aqueous) increased from 0.05 at an acid concentration of 0.2 *N* to 4.3 at 10.2 *N* in the absence of uranium. The addition of 1.0 *M* $NaNO_3$ increased the extraction of plutonium IV by a factor of 2 while 4.0 *M* $NaNO_3$ increased the extraction coefficient by a factor of 8 (Table 10-11).

The distribution coefficients of Pu III and Pu VI were apparently less than that of Pu IV, but the data were somewhat erratic and are being rechecked.

As the concentration of uranium in the aqueous phase was increased from 0.1 to 0.5 *M*, the extraction of both Pu IV and Pu VI was decreased by factors of five to ten (Table 12).

Solvent Diluents. In early work with tributyl phosphate it was found necessary to use a diluent because of the high specific gravity and viscosity of tributyl phosphate. A diluent was also necessary in this process for two additional reasons; one, when a lean mixture (< 20%) solvent was saturated with uranium, the extraction of fission products was at a minimum; two, the uranium distribution coefficient for stripping was dependent on the concentration of TBP in the solvent mixture and the distribution coefficient (A/O) dropped below 1 for concentrations greater than 20% TBP. Thus a mixture of 15% tributyl phosphate-85% diluent was chosen as an acceptable composition for the process solvent.

TABLE 10

Effect of Acid Concentration on TBP Extraction of Ce III, Zr, and Pu IV

Aqueous Phase: 0.1 *M* $UO_2(NO_3)_2$ tracer, nitric acid

Organic Phase: One equal volume pass 15% TBP in hexane

HNO_3 CONCENTRATION (Molarity)	DISTRIBUTION COEFFICIENTS (org/aq)		
	Ce III Beta	Zr Beta	Pu IV Alpha
0.2	ca. 0.001	0.004	0.05
1.1	ca. 0.001	0.007	0.23
2.7	ca. 0.001	0.16	2.04
6.1	ca. 0.001	0.267	5.44
10.2	ca. 0.001	0.716	4.38

TABLE 11

Effect of Sodium Nitrate Concentration on TBP Extraction of Ce III, Zr, Pu IV

Aqueous Phase: 0.1 *M* $UO_2(NO_3)$, 3.0 *M* HNO_3 $NaNO_3$, tracer

Organic Phase: One equal-volume pass 15% TBP in hexane

$NaNO_3$ CONCENTRATION (Molarity)	DISTRIBUTION COEFFICIENTS (org/aq)		
	Ce III Beta	Zr Beta	Pu IV Alpha
0	ca. 0.003	0.16	2.04
0.1	ca. 0.003	0.17	2.1
0.6	ca. 0.003	0.25	4.1
1.0	ca. 0.003	0.26	4.4
3.0	ca. 0.003	0.61	9.7

TABLE 12

Effect of Uranium Concentration on Plutonium Distribution Coefficients (O/A)

Aqueous Phase: 1.0 *N* $NaNO_3$ - 3.0 *M* HNO_3; $UO_2(NO_3)_2$

Organic Phase: 15% Bu_3PO_4 in hexane, one equal volume pass

Pu VALENCE STATE	D.C. O/A AT 24 g U/*l*	D.C. O/A AT 70 g U/*l*	D.C. O/A AT 120 g U/*l*
1. Pu III	2.0	0.50	0.24
2. Pu IV	3.5	0.7	0.26
3. Pu IV	-	0.7	-
4. Pu VI	1.0	0.2	0.06

1. Pu III Distribution—tracer reduced with $Fe(NH_4)_2(SO_4)_2$ and 2 *N* $H_2OH \cdot H_2SO_4$; solution 0.05 *M*-2 *N* $H_2OH \cdot H_2SO_4$

2. Pu IV Distribution—tracer as received in 1 *N* HNO_3; no oxidizing or reducing agents added.

3. Solution made 0.1 *M* in $NaNO_2$.

4. Pu VI Distribution—tracer oxidized with $Ce(SO_4)_2 \cdot (NH_4)_2SO_4$; solution 0.05 *M* $Na_2Cr_2O_7$

In most of the experimental work thus far, hexane has been used as the diluent. However, it has the undesirable properties, of a low flash point and high volatility. Varsol has been found to be an acceptable substitute and has been chosen as the most desirable of those yet investigated.

From the results of four countercurrent batch runs made with ORNL sludge solution under identical conditions using both hexane and "Varsol" as diluents, it was concluded that there was no difference in the separation of uranium from fission products, or in uranium extraction and stripping with either hexane or "Varsol" as diluent for tributyl phosphate (Table 13).

Solvent Treatment. Both carbonate and caustic washes of used (85% hexane-15% TBP) solvent showed significant improvement on the uranium losses, but little effect on decontamination was in countercurrent batch comparison runs.

With 12% varsol-88% TBP no apparent deterioration of the solvent was seen from prolonged contact with 5 *N* acid feed solution; however, a 1 *M* Na_2CO_3 wash treatment is recommended to remove residual activity and color from the used solvent.

Effect of Uranium Concentration in Extraction Feed. Uranium solutions as dilute as 24 g/*l* were extracted successfully with tributyl phosphate in "Varsol" with adequate decontamination by decreasing the solvent to feed flow ratio and maintaining a 70% uranium saturation of the organic phase at the feed plate. This degree of saturation can be obtained by decreasing the scrub acidity, allowing higher uranium reflux from the scrub section (Table 14).

The feeds extracted the countercurrent batch equipment as described below ranged from 24 to 100 grams of uranium per liter.

In the first run, F-1, with 6½% TBP the extraction factor was too low to effect complete extraction in five stages. This run was repeated (F-2) using 13% TBP and water as a scrub to increase the uranium concentration in the solvent sufficiently to give acceptable decontamination. It is possible that, due to the high reflux of uranium in the scrub section, this run did not reach complete equilibrium; however, considerable improvement can be seen.

In runs F-3 and F-4 the IAW losses were greater than 1% even with seven extraction stages. This loss could be reduced by increasing the TBP concentration in the solvent or increasing the solvent flow rate to increase the extraction factor. At the same time it would be necessary to increase the uranium reflux as in run F-2 to effect decontamination.

The strip from F-5 was concentrated and analyzed for Ru and Zr-Cb. Fifty percent of the activity was Ru and 26% was Zr-Cb.

TABLE 13

Comparison of Varsol and Hexane as a Diluent for Tributyl Phosphate Counter-Current Batch Extraction

ORNL WASTE METAL ACTIVITY LEVEL

FEED:
U = 118.0 mg/ml
HNO_3 - 3.4 *N*
β - 3.2×10^6 c/m/ml

SOLVENT:
15% - $(C_4H_9)_3 PO_4$
85% - Diluent

SCRUB:
3.0 HNO_3

STRIP:
Dist. H_2O

FLOW RATES: Org.: 100 ml FEED: 30 ml SCRUB: 20 ml STRIP: 100 ml

DILUENT	BETA FISSION PRODUCTS			URANIUM LOSSES (Percent)	
	c/m/mg - U IAF	c/m/mg - U IBP	Decontamination Factor	IAW	IBW
Varsol	2.7×10^4	1	10^4	0.10	0.39
Hexane	2.7×10^4	1	10^4	0.09	0.53

HW WASTE METAL ACTIVITY LEVEL

FEED:
U - 114.0 mg/ml
HNO_3 - 3.4 *N*
β - 4.2×10^7 c/m/ml
I.C. 250 mv/ml

SOLVENT:
15% - $(C_4H_9)_3 PO_4$
85% - Diluent

SCRUB:
3.0 HNO_3

STRIP:
Dist. H_2O

FLOW RATES: Org: 90 ml FEED: 30 ml SCRUB: 20 ml STRIP: 90 ml

DILUENT	BETA FISSION PRODUCTS			GAMMA ACTIVITY OF IBP	URANIUM LOSSES (Percent)	
	c/m/mg - U IAF	c/m/mg - U IBP	Decontamination Factor		IAW	IBW
Varsol	3.7×10^5	5	7×10^4	Background	0.14	0.55
Hexane	3.7×10^5	3	1×10^5	Background	0.28	0.27

TABLE 14

Effect of Varying Uranium Concentration in TBP Waste Metal Recovery Runs

F-1,2 Acidified HW Supernatant (103-U)
F-3,4 HW supernatant precipitated, settled and dissolved
F-5,6 HW supernatant precipitated, centrifuged, washed and dissolved

Feed: Scrub ratio = 2:1 in all runs.
Solvent rate calculated to give ca. 70% uranium saturation at the feed plate.

RUN NO.	FEED			STRIP (IBP)		μ LOSSES (Percent)		SCRUB	SOLVENT	STAGES		
	g U/l	M HNO_3	β c/m/mg U	β c/m/mg U	β D.F.	IAW	IBW	M HNO_3	%$(C_4H_9)_3PO_4$	Ext.	Scrub	Strip
F-1	24	2.7	3.8×10^5	7.8	5×10^4	8.5	0.00	3.0	6.5	5	4	5
F-2	26	4.4	3.2×10^5	8.6	4×10^4	0.21	0.21	0.0	13	6	4	4
F-3	39	2.9	2.5×10^5	3.2	8×10^4	3.1	0.01	4.5	10	5	4	5
F-4	39	2.0	2.8×10^5	2.6	1.1×10^5	1.5	0.07	3.0	10	7	4	3
F-5	100	3.2	1.7×10^5	18.1	1.0×10^4	0.07	0.00	3.0	15	6	4	4
F-6	75	4.6	1.3×10^5	3.9	4×10^4	0.04	0.05	3.0	12	5	4	5

COLUMN STUDIES

Process Demonstration. Seven runs were made in the one inch column at flowsheet conditions to demonstrate the performance and operability of the TBP metal recovery process. The feeds for these seven runs were nitric acid solutions of ORNL metal waste sludge.

The beta fission product activity of the recovered uranium from all these runs was less than 10% of the activity of natural uranium, and with six feet of scrub section the activity of the product was not significantly different from the background of freshly extracted uranium. In Run 21 the uranium concentration in the solvent at the feed plate was only 30% of saturation which resulted in a drop in decontamination. However, the activity of the product was still less than 10% of the activity of natural uranium (Table 15).

The IAW uranium loss was about 0.1% with the 10 feet of extraction section used. A longer extraction section would not significantly reduce this loss since approximately this amount of uranium has been found to be inextractable from ORNL sludge solutions.

The IBW uranium loss was about 0.2% from the 9 packed feet of strip column. This loss can be reduced to any desired value by additional column height or increased flow of IBX.

Run 24 demonstrated the recovery of uranium from a solution of ORNL sludge which contained only 66 *g/l* of uranium. This was accomplished by reducing the organic to aqueous flow ratio to 1.5:1 and reducing the concentration of TBP in the solvent mixture to 12%.

Run 25 demonstrated the recovery of uranium from HW supernatant. The IAF for this run was prepared by precipitating the uranium from HW supernatant by the addition of an excess of NaOH. The sludge thus formed was then centrifuged and the cake dissolved in 70% HNO_3 to an excess of 4.5 *N* in the final IAF. The uranium recovered from this feed contained 7 beta fission product counts per minute per milligram of uranium and the total uranium loss was 0.2%.

Flooding Rates for IA and IB Columns. The flooding rate for the IA column was found to be 1000 gal/hr/ft^2 total through-put. The flooding rate for the IB column was found to be 500 gal/hr/ft^2 total through-put.

Flooding rates were determined in a 1½ inch pyrex glass pipe column packed with 1/4 × 3/8 inch split rings. The column was 10 feet in length and contained about 9 feet of packed section. No scrub section was used, and the IAF and IAS were mixed before entering the column.

TABLE 15

Summary of Waste Metal Recovery Column Runs

COLUMNS: One inch pyrex glass pipe, packed with ¼ × ¼ Raschig rings.

1A; Extraction length = 10 ft = Scrub length as given in table

1B; About 9 ft packed section

FEEDS:

1AF; Solution of ORNL sludge with 70% HNO_3 to about 3 *N* excess acid

1AS; (3 *N* HNO_3)

1AX; $(C_4H_9)_3 PO_4$ diluted with "varsol" to concentration indicated

1BX; water

FLOW RATIOS:

IAF; IAS; IAX; IBS = 2: 1: ca6: ca6

RUN NUMBER	LENGTH OF SCRUB SECTION	$(C_4H_9)_3 PO_4$ CONCENTRATION PERCENT	PERCENT SATURATION AT FEED PLATE	BETA ACTIVITY OF IAF c/m/mg U	BETA ACTIVITY OF IBP c/m/mg U	β D.F.	URANIUM LOSS (Percent) IAW	IBW
18	4	15	60	2.3×10^4	4	6×10^3	0.07	0.2
19	4	15	66	2.6×10^4	4	6×10^3	0.07	0.2
20	4	15	60	2.6×10^4	6	4×10^3	0.08	0.2
21	6	15	30	2.7×10^4	4	7×10^3	0.11	0.2
22	6	15	50	2.7×10^4	< 1	> 1×10^4	0.06	0.2
23	6	15	66	2.7×10^4	< 1	> 1×10^4	0.13	0.1
24	6	12	47	2.2×10^4	< 1	> 1×10^4	0.05	0.2
25*	6	12	66	1.2×10^5	7	1.7×10^4	0.11	0.09

* The metal feed solution for Run 25 was prepared from HW supernatant by precipitation of the uranium with an excess of NaOH, centrifugation and dissolution of the precipitate in 70% HNO_3 to about 5 *N* excess acid. The resulting solution contained 68 g/*l* of uranium.

TABLE 16

Uranium Concentration Data for HETS Determination

COLUMN: 1½ inch glass pipe, packed to a height of 9 ft with 1/4 x 3/8 inch Raschig rings.

AQUEOUS FEED: Run 1

Mixture of one part 3.9 *N* HNO_3 and two parts HNO_3 solution of sludge from W-10, ORNL; 120 g/*l* U, 3.0 *N* acid.

Runs 2, 3 and 4

Mixture of one part 3.0 *N* HNO_3 and two parts synthetic ORNL sludge solution; 1.0 *M* $NaNO_3$, 0.7 *M* Na_2HPO_4, 0.8 *M* Na_2SO_4, 0.6 *M* UNH

SOLVENT: 85% "varsol", 15% $(C_4H_9)_3PO_4$

RUN CODE NO.	AQUEOUS URANIUM CONCENTRATION (g/*l*) AT VARIOUS DISTANCES BELOW FEED PLATE (feet)						
	Feed Solution	1.3	2.8	4.3	6.8	9 Ft. IAW	IAP
1	72	22	7.0	2.4	.40	.12	31
2	95	23	8.5	2.5	.35	.22	44
3	95	19	4.8	1.1	.16	.07	43
4	95	20	4.0	1.4	.15	.05	35

TABLE 17

Counter-Current Batch Extractor Runs for Uranium Data

FEEDS: Run 1
Same as feed for Column Run 1, Table 16

Run 2
Same as feed for Column Runs 2, 3, 4, Table 16

SOLVENT: 15% $(C_4H_9)_3PO_4$ - 85% varsol

EXTRACTOR: Four tubes

FLOW RATES: Run 1 Feed = 36 cc/batch, solvent = 67 cc/batch Run 2 Feed = 36 cc/batch, solvent = 82 cc/batch

RUN NUMBER	URANIUM CONCENTRATION (g/l)							
	FIRST TUBE		SECOND TUBE		THIRD TUBE		FOURTH TUBE	
	Aq. Phase	Org. Phase	Aq. Phase	Org. Phase	Aq. Phase	Org. Phase	Aq. Phase	Org. Phase
1	17	38	3.3	9.2	0.13	0.44	0.06	0.02
2	27	42	2.5	12	0.12	0.53	0.02	0.03

The flooding rate was the rate of flow at which the packing no longer broke up the dispersed phase, or at which a second interface formed at the bottom of the column. For the IA column, flooding was found to occur essentially at the same total through-put for two flow ratios; solvent: aqueous phase = 1:1 and solvent: aqueous phase = 2:1. Only one flow ratio was tested for the IB column; solvent: aqueous phase = 1:1.

HETS Determination. Average HETS values for a 1½ inch column 9 feet in length and packed 1/4 × 3/8 inch Raschig rings were: 75% of flooding—3.6 feet, 50% of flooding—3.0 feet, and 25% of flooding—2.5 feet.

HETS determinations were determined in a 1½ inch column, 9 feet in height packed with 1/4 × 3/8 inch split rings and having four samplers set into the column in such a way as to permit sampling of the aqueous phase at four intermediate points in the column. Four such runs were made, one using ORNL sludge solution and three using a synthetic feed of approximately ORNL sludge solution composition.

With synthetic feed runs made at 25%, 50% and 75% of flooding, the column was operated for more than three complete changes of the aqueous phase. Duplicate samples were taken for uranium analysis of the feed solution, of the aqueous phase at the four intermediate heights along the column, and of the aqueous raffinate. The run with ORNL sludge solution was made at about 60% of flooding and the same procedure followed.

In connection with the column runs, the same solutions were processed in a four-stage counter-current batch extractor using the same flow ratio that was used in the column. The tubes were sampled after three complete changes and the uranium in both phases determined.

Figure 1 is a conventional XY diagram constructed from the data obtained from the column run made with ORNL sludge solution. The equilibrium line is plotted from the data obtained from a four tube counter-current batch extractor run made with the identical solutions. By this method average HETS values were found to be as follows: (ORNL sludge solution) 60% of flooding—3.0 feet; (synthetic sludge solution) 25% of flooding—2.5 feet, 50% of flooding—3.0 feet, and 75% of flooding—3.5 feet.

Figure 2 shows a plot of the uranium concentration (grams/liter) in the aqueous phase as a function of distance from the feed plate (feet). The curve given was determined with synthetic ORNL sludge solution processed in the column at 50% of flooding. The HETS values for each stage were then determined by plotting the concentration of uranium in the aqueous phase of each tube of

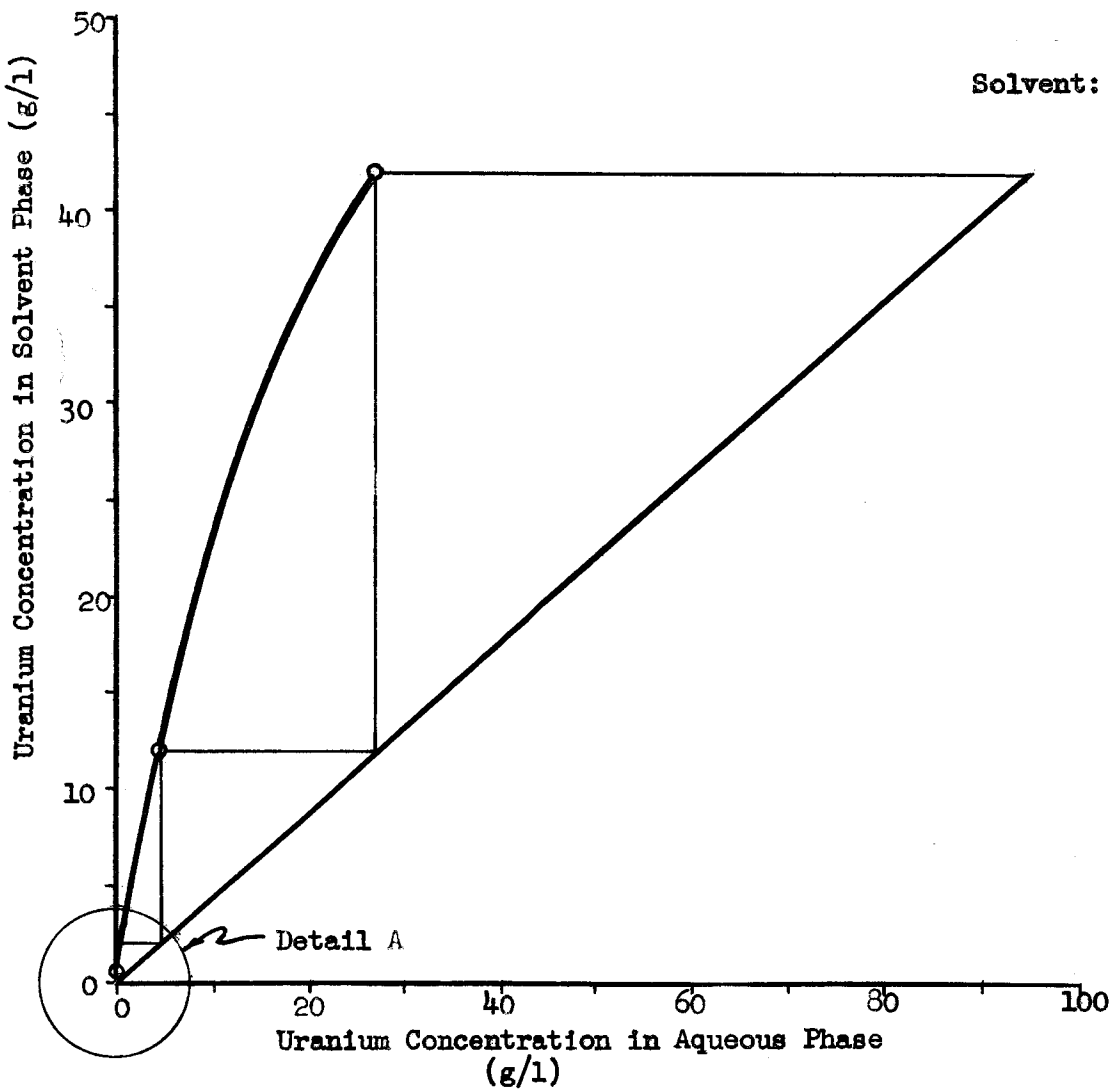
Figure 1

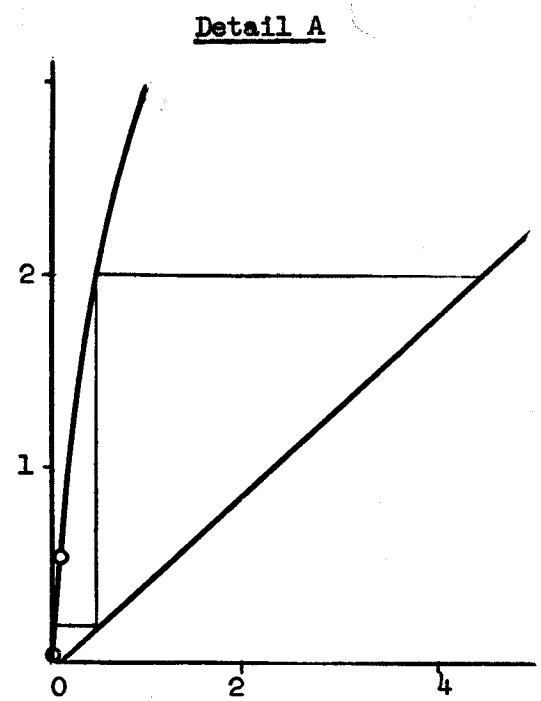
X-Y Diagram for ORNL Waste Metal Recovery

Run made at 50% of flooding in a 1-1/2 inch column 9 ft. in length, packed with 1/4x3/8 inch Raschig rings

Aqueous Feed: 95 g/l of Uranium
0.5 M Na_2SO_4
0.5 M $Na_2H\ PO_4$
0.6 M $NaNO_3$
3.0 M HNO_3

Solvent: 85% Varsol - 15% $(C_4H_9)_3PO_4$





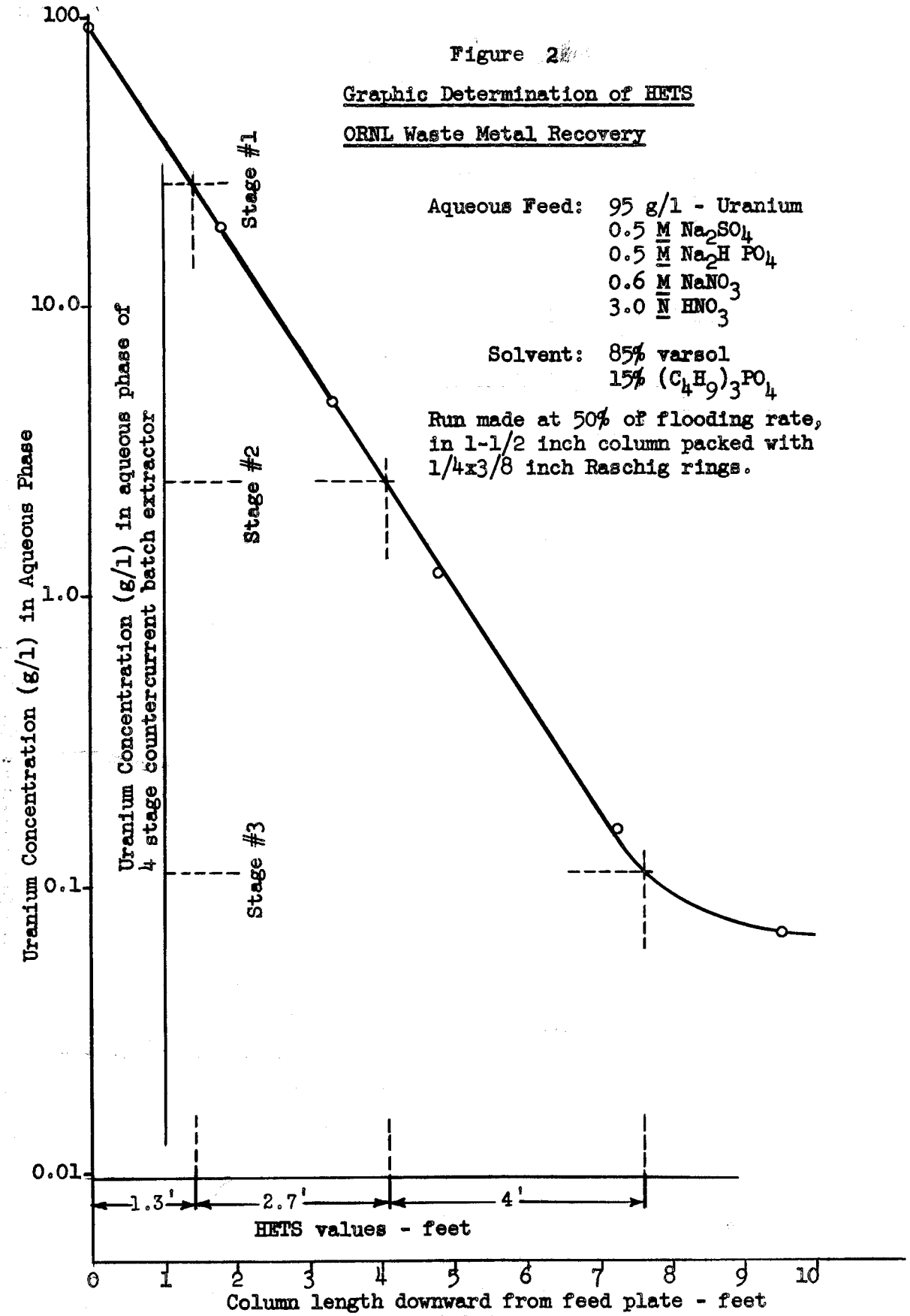


Figure 2

Graphic Determination of HETS

ORNL Waste Metal Recovery

Aqueous Feed: 95 g/l - Uranium
0.5 M Na_2SO_4
0.5 M Na_2HPO_4
0.6 M $NaNO_3$
3.0 N HNO_3

Solvent: 85% varsol
15% $(C_4H_9)_3PO_4$

Run made at 50% of flooding rate, in 1-1/2 inch column packed with 1/4x3/8 inch Raschig rings.

the four stage counter-current batch extractor made with identical conditions, determining where these concentrations intercept the uranium concentration line of the column, and scaling off the HETS from these intercepts. (Table 18).

The Effect of Scrub Length on Decontamination. Increased length of scrub section gave a marked improvement in the beta decontamination factor. The decontamination factor was 6×10^2 with no scrub section, 8×10^3 with four feet of scrub section and 1×10^4 with ten feet of packed scrub section (Table 19).

FEED PREPARATION

Development of uranium feed preparation for the TBP solvent extraction process includes removal of the uranium-bearing sludge from underground retention tanks, and preparation of a clarified acid solution of the uranium (0.5 *M* uranium, 3.0 *N* nitric acid) to be fed to the TBP solvent extraction columns.

The principal steps in preparing feed material are:

(1) transfer of uranium solution and sludge from retention tanks to processing units,
(2) concentration of uranium slurries to about 35% solids by volume,
(3) acid dissolution of the solids,
(4) clarification of the acid solution to remove all insolubles.

A centrifugal sump pump, immersed in the sludge layer of Tank W-10 in the ORNL Tank Farm, delivered a fairly concentrated material, i.e., about 35% solids by volume. Average slurry concentration as pumped is expected to be about 20-25% solids by volume. Use of a boom-type arm for maneuvering the pump's suction line over the entire tank bottom was suggested, and is being developed by the Design Group.

Possible concentration methods include filtration, centrifugation, continuous sedimentation, and evaporation. To date, major emphasis has been placed on evaluating filtration as a method for concentrating the slurry. The results of this work indicate that a pre-coat type rotary filter may be satisfactory.

By using an experimental filter stick with 700 × 60 mesh stainless steel screen and with a precoat of diatomaceous earth filter aid but no filter aid in the slurry, a high initial flow rate was obtained. Using 16 in. Hg vacuum,

TABLE 18

HETS for Tributyl Phosphate Extraction of Uranium

(Determined by Graphic Method Illustrated in Fig. 2)

COLUMN: 1½ inch Pyrex glass pipe, packed with 1/4 x 3/8 inch Raschig Rings about 9 ft of packing.

SOLVENT: 85% varsol, 15% $(C_4H_9)_3PO_4$

FEED: Run 1
Mixture of one part of 3.0 *N* HNO_3 and two parts of HNO_3 solution of sludge from ORNL - W-10, 120 g/*l* - U, and 3.0 *N* acid.

Run 2, 3, 4
Mixture of one part of 3.0 *N* HNO_3 and two parts synthetic ORNL sludge solution; 3.0 *N* HNO_3, 1.0 *N* $NaNO_3$. 0.7 *M* Na_2HPO_4, 0.8 *M* Na_2SO_4 and 0.5 *M* UNH.

RUN CODE NO.	PERCENT OF FLOODING RATE	LENGTH OF FIRST STAGE (feet)	LENGTH OF SECOND STAGE (feet)	LENGTH OF THIRD STAGE (feet)
1	60%	2.0	2.5	4.5
2	75%	1.6	2.8	Less than 3 complete stages
3	50%	1.3	2.7	4.0
4	25%	1.3	2.7	3.6

TABLE 19

Effect of Scrub Length on Decontamination

COLUMN: One inch glass pipe
Scrub Section as noted in Table
Strip Section 10 feet

FEED: ORNL waste sludge solution from W-10. 3 *N* HNO_3 flow rate 15 cc/min.

SOLVENT: 85% hexane—15% tributyl phosphate, flow rate 45 cc/min.

SCRUB: 3 *N* HNO_3 flow rate 7.5 cc/min.

STRIP: Demineralized water, flow rate 45 cc/min.

SCRUB SECTION LENGTH (feet)	EXTRACTION SECTION LENGTH (feet)	OVERALL BETA DE-CONTAMINATION FACTOR	IAW U LOSS (Percent)
0	10	6×10^2	0.05
4	10	8×10^3	0.07
10	4	1×10^4	1.4

the average flow rate was 1.0 gal/sq ft/hr for the first five minutes, for 25% solids slurry. The flow rate dropped off very quickly to zero, which fact dictates a short filtration cycle of less than 10 minutes for a continuous (rotary) operation.

By using an experimental cup filter and a filter stick with 700 × 60 mesh stainless steel screen and with diatomaceous earth filter aid in the slurry, average flow rates were 0.5 gal/sq ft/hr for the first thirty minutes. Based on this flow rate, 35 sq ft of active filtering area would be required to concentrate feed slurry from 21% solids by volume to 35% solids. From viscosity considerations this filtration rate can be approximately doubled by raising the temperature 75° F. The effect of different types and quantities of filter aid was slight (Table 20). Filtration equations are presented in Table 21.

From preliminary investigations, it appears that centrifugation as a method of feed concentration is feasible. A 16% solids slurry of W-10 material, fed at a flow rate of 600 ml/min into a Sharples Super Centrifuge developing a centrifugal force of 6000G, yielded a clear effluent. There are commercially available machines, which, with some modifications, could handle such material.

Sedimentation trials to date indicate that the rate of increase of solids concentration in percent by volume is of the order of 2% per day. This suggests that continuous sedimentation might involve a two week hold-up of material in a thickener tank of about 30,000 gallons capacity, and therefore sedimentation under these conditions is impractical. Other conditions (i.e., higher temperature, etc.) under consideration may enhance sedimentation rates.

Two evaporation trials made by first dissolving slurry from W-10 with nitric acid and then evaporating to the desired uranium concentration (0.5 molar) indicated that crystallization will commence at roughly 0.4 molar uranium. Unless crystallization can be prevented by washing the sludge in W-10 with water, evaporation does not appear too attractive.

Dissolution of the sludge may be accomplished with nitric acid, but the rate is limited by the evolution of carbon dioxide. In the tank farm set-up, which has insufficient agitation, roughly 100 gallons of sludge was dissolved in 3 to 4 hours. Rapid and thorough agitation is necessary to facilitate dissolution without violent foaming.

Clarification of the acid solution of uranium to remove siliceous and other foreign material can be accomplished by filtration using 0.5 to 2 grams of Celite 545 diatomaceous filter aid per 100 ml of slurry and a 700 × 60 mesh stainless steel screen precoated with ¼ in. of Celite 545. Apparently, the

TABLE 20

Constant Pressure Filtration of Tank W-10 Sludge

Filter Medium:	700 and 60 mesh stainless steel screen
Diameter of Cup Filter:	3 in.(Runs 7-27)
Diameter of Filter Stick:	3 in. (Runs 23A-40)
Particle Size:	1-5 microns
Viscosity of Supernatant Liquid (80° F):	3 cps
Density of Supernatant Liquid:	1.13
Density of Dry Solids:	1.90
Temperature:	25 - 30° C

TRIAL	SLURRY		PRECOAT	VACUUM (inches of mercury)	AVERAGE FLOW RATE (gal/ft^2 hr) FOR FIRST							REMARKS
	Solids	Grams of filter aid per 100 ml			3 min.	8 min	15 min	30 min	45 min	70 min	90 min	
7	21%	5g Hyflo Super Cel	¼ in.Celite 545	4	0.70	0.65	0.56	0.39	0.30	---	---	
8	21%	5g Hyflo Super Cel	¼ in.Celite 545	4	---	0.24	0.29	0.27	0.22	---	---	No filtrate entered receiver until 3 min after start.
9	21%	5g Filter Cel	¼ in.Celite 545	8	1.17	0.79	0.63	0.45	0.33	---	---	
10	21%	2½ g Hyflo Super Cel	¼ in.Celite 545	16	1.0	0.70	0.56	0.43	0.39	---	---	
10A	21%	2½ g Hyflo Super Cel	¼ in.Celite 545	16	0.70	0.61	0.54	0.43	0.37	0.31	---	
11	21%	2½ g Hyflo Super Cel	¼ in.Celite 545	8	0.70	0.57	0.49	0.37	0.32	0.25	---	
26	21%	2½ g Stan. Super Cel	None	8	1.22	0.69	0.49	0.36	0.254	0.246	---	28% solids thinned to 21% by addition of water
27	21%	2½ g Stan. Super Cel	None	16	1.47	0.97	0.69	0.48	0.41	0.30	0.25	
23A	25%	2½ g Stan. Super Cel	None	4	1.35	0.81	0.56	0.37	0.27	---	---	
34	25%	2½ g Stan. Super Cel	None	8	1.4	0.78	0.56	0.41	0.33	0.27	0.24	
35	25%	2½ g Stan. Super Cel	None	16	1.9	1.1	0.79	0.54	0.44	---	---	
39	25%	2½ g Filter Cel	None	8	1.3	0.79	0.58	0.42	---	---	---	
40	25%	2½ g Filter Cel	None	16	2.2	1.15	0.79	0.53	---	---	---	23A-40 Filter Stick

All runs at room temperature

No. 7 - No.27 Cup Filter

No. 23A No. 40 Filter Stick

TABLE 21

Filtration Equation for Tank W-10 Sludge

CODE: P - pressure drop across filter
V - total volume of filtrate
A - filter area
θ - time after start of filtration

EQUATION P - in. Hg V - gal A - ft^2 θ - hr	CONDITION OF FILTRATION—SLURRY		PRECOAT	APPROXIMATE RELIABILITY OF EQUATIONS		
	Solids (Percent)	Filter aid/100 ml		Range of θ (min)	Range of P (in.Hg)	Max deviation of V from obs values
$(P\theta)/(V/A) = 24.8\ P^{0.523}\ (V/A) + 2.55\ P^{0.598}$	21	2½ g Hyflo Super Cel	¼ in.Celite 545	10-90 10-120	8-16 8-18	3% 5%
$V/A = 0.132\ \theta^{1/2}\ P^{3/8}$	25	2½ g Standard Super Cel	None	10-90 10-90 10-120	8-16 4-18 8-18	5% 15% 10%
$V/A = 0.155\ \theta^{1/2}\ P^{1/3}$	25	2½ g Filter Cel	None	10-35 10-60	8-16 8-16	6% 10%
$V/A = 0.115\ \theta^{1/2}\ P^{0.387}$	21	2½ g Standard Super Cel	None	10-90 10-40	8-16 8-16	11% 6%

ease with which the dissolved slurry filters decreases with age. Filtration of a month old slurry to which ½ gram of Celite 545 per 100 ml was added, at a vacuum of 5.4 in. Hg yielded an average flow rate of 90 gal/sq ft/hr for the first twenty-five minutes (Table 22). A centrifugal clarifier would also serve in this step as evidenced by the rapid clarification of samples of acid solution in a serum centrifuge.

EQUIPMENT INSTALLATION FOR HANFORD METAL WASTE SEMI-WORKS DEVELOPMENT

Equipment for development of the TBP metal waste extraction process is being installed in two 6 ft × 6 ft × 30 ft cells in Building 706-HB. All essential equipment items are on hand, except for a few special items on which delivery by September 21 is promised.

The dimensions of the two-column solvent extraction system are:

	INTERNAL DIAMETER	HEIGHT OF PACKED SECTION
Extraction Column—Extraction Section	1.61 in.	12.5 ft.
Extraction Column—Scrub Section	1.61 in.	9.8 ft.
Strip Column	2.47 in.	18 ft.

The strip column diameter was increased because the strip column flooding rate was found to be 500 gal/hr/sq ft compared to 1000 gal/hr/sq ft for the extraction column. The strip column uranium loss goes up rapidly as the number of strip stages is reduced, so the diameter of the strip column was proportioned to the extraction column diameter, permitting both columns to operate at the optimum flow rate for maximum stage efficiency.

TABLE 22

Filtration of Dissolved Sludge from Waste Tank W-10

Filter Area: 0.44 sq in., 2.85 sq cm

Per cent solids less than 0.5%

TRIAL	FLOW RATE ($ml/cm^2 min$) AT			AVERAGE FLOW RATE FOR FIRST 25 MIN (ml/cm^2 min)	PRESS. DROP ACROSS FILTER	PRECOAT	FILTER AID PER 100 ml SLURRY	DATE OF TEST
	6 min	12 min	25 min					
14	0.63	--	--	--	15 in. H_2O	1/4 in. Celite 545	None	7-19-49
21	5.40	3.6	2.4	3.6	5.4 in. Hg	1/4 in. Celite 545	2 g Celite 545	7-21-49
22	6.9	4.6	2.7	6.0	5.4 in. Hg	1/4 in. Celite 545	½ g Celite 545	7-21-49
28A	1.9	0.5	--	--	2.4 in. Hg	1/4 in. Celite 545	2 g Celite 545	8-3-49
30-31 Ave.	2.2	0.7	0.05	2.0	5.4 in. Hg	1/4 in. Celite 545	2 g Celite 545	8-4-49
33	1.5	0.5	0.25	1.7	5.4 in. Hg	1/5 in. Celite 545	½ g Celite 545	8-4-49
36	0.28	0.12	0.02	0.34	2.4 in. Hg	1/4 in. Hyflo S.C.*	½ g Hyflo S.C.	8-5-49
37	0.49	0.22	0.05	0.51	5.4 in. Hg	1/4 in. Hyflo S.C.	½ g Hyflo S.C.	8-5-49
38	1.4	0.3	0.1	1.5	5.4 in. Hg	1/4 in. Celite 545	1 g Celite 545	8-5-49

All Runs at room temperature

* S.C. = Super Cell

RADIOCHEMICAL WASTE PROCESSING

WASTE COLLECTION AND MONITORING SYSTEM

The proposals for altering and expanding the existing ORNL liquid waste collection system as outlined in previous quarterly reports have now been approved and incorporated as a part of the plans for the permanent Laboratory. The Plant Engineering Department is making construction drawings for those liquid waste system alterations within the confines of the existing Laboratory Area. New collection tanks for radiochemical and metal waste solutions have been located. The Austin Company is studying the installation of liquid waste lines from the new Research, Semi-Works, and Isotope Research Buildings.

Since the expansion of the Laboratory is occurring to the east of the existing plant site and downgrade from the existing tank farm, the length of transfer lines required to connect new buildings to the existing waste facilities is quite long, and an appreciable gravity head must be overcome by transfer devices. For this reason two investigations have been started:

1. A jet testing station is being installed in Building 9204-1, Y-12, to collect jet data useful in the selection of proper transfer devices. Since the required head for transfer devices on waste collection and monitoring tanks in the new research areas may exceed that available from steam jets, an investigation of pumps for handling radioactive liquids will be prosecuted. To start this investigation, a LaBour Type "G" self priming packless pump of stainless steel has been ordered.

2. The cost of underground transfer lines constructed of stabilized stainless steel is exceptionally great for the long lines required in the Laboratory expansions. The use of satisfactory, cheaper substitutes for stainless, such as polythene lined, acid resistant concrete pipe is being investigated.

LIQUID WASTE EVAPORATOR OPERATION

The evaporator was accepted from the J. A. Jones Construction Company on May 27, 1949. Prior to acceptance, the evaporator tanks were calibrated and intensive leak checking was carried out. The first test run was made using synthetic feed approximating the chemical composition of the tank farm wastes

with added tracer activity. Hot runs using waste from W5 were started immediately after the first test run as the underground waste storage tanks were filled and overflowing to the settling basin. This situation has been remedied to the extent that the volume of liquid in W5 has been drastically reduced, and wastes from other storage tanks are now being evaporated. A design report for the evaporator, ORNL-393, will be issued soon.

Evaporator Heat Transfer. Overall coefficients of heat transfer were calculated for the first seven runs. From these results and operating data, the following conclusions may be made:

1. No detectable scaling of the heat transfer surfaces has occurred during 600 hours of operation.

2. The average coefficient of heat transfer is 386 Btu/hr/sq ft/°F at an average Δt of 43° F. A somewhat better correlation of the data is obtained by assuming all transfer area is submerged. The average overall heat transfer unit, U, for this assumption is 300 Btu/hr/sq ft/°F.

3. The geometric arrangement of the heating surface and possibly foaming, combine to partially blanket the upper portion of the transfer surface with vapor bubbles. This is evidenced by a decrease of U with increasing Δt for both the partially and completely submerged surface. This effect has not hindered operations.

4. An appreciable increase in boiling point occurs during a run.

5. The steam consumption of the evaporator is unknown.

Condenser Heat Transfer. The four condensers are adequately sized for normal evaporation rates and are just sufficient to condense all vapor at the maximum water vaporization rate of about 800 gallons/hr. Vapor distribution from the manifold to the four condensers is unequal, causing the two outside condensers to carry about 75% of the load. Cooling water rates were set so the two end condensers receive more water and any possibility of vapor blowby was eliminated.

Appreciable subcooling of the condensate occurs in the condenser tubes. This makes it impractical to estimate overall condensation coefficients during normal operation as the actual area for condensation is unknown. A special test run was made in which the cooling water flow was reduced so the exit condensate was almost boiling (~ 209° F). For this condition of negligible subcooling, the overall heat transfer coefficient, U, for the four condensers as a unit was calculated to be 233 Btu/hr/sq ft/°F. The total vapor lead was 3750 lbs/hour and the total cooling water flow, from the temperature rise of

98.4° F, was 37,300 lbs/hr. Condensation coefficients in operation are larger than this as faster cooling water flows are used. The experimental coefficient of U = 233 Btu/hr/sq ft/°F may be compared with U = 158 Btu/hr/sq ft/°F calculated from theoretical water and steam film resistances. No appreciable fouling of condensers has been observed.

Decontamination. To date (June 31, 1949), one preliminary tracer run using synthetic waste solution and eight complete 100% activity level runs have been made. The operating results for all runs are presented in Table 23, and radiochemical analysis of the feed and condensate for several runs are given in Table 24. Ionic analysis of the sludge composite from two runs is given in Table 25. The overall decontamination factor is defined as the ratio of the feed to condensate gross β activities. Material recycled to W5 because of foam-over contamination was not included in the condensate.

Analytical difficulties reduced the extent of the investigation. However, the following conclusions may be made:

1. The cyclone separator gives an average decontamination factor of 3.1.

2. Mechanical entrainment during normal operation is in the range observed in commercial evaporators. Exclusive of iodine, about 15 parts per million parts evaporated were carried over by droplets into the condensate at the midpoint of Run 4. The total entrainment before the cyclone separator is about 47 parts per million parts evaporated.

3. The major contributor to the condensate activity in Runs 4 and 9 is iodine regardless of the evaporator *p*H. Appreciable volatilization of iodine occurs, even from basic solutions.

4. The feed for Runs 6 and 7 was long cooled material. This, and the high *p*H feed reduced the amount of iodine carry-over to the range of entrainment carry-over.

5. The ratios of Sr, Ru, Cs, and TRE to each other in the feed and condensate are approximately the same, indicating that there is no selective volatilization of any of these radioelements.

6. Overall decontamination factors > 1000 are reproducible in the absence of foaming.

Evaporator Foaming. The only serious operating problem is evaporator foaming, which assumes the nature of a violent eruption at times. Foaming reduces the decontamination factor by carrying unevaporated, concentrated liquid waste from the evaporator to the condensate tank. Foaming occurred in all runs, but the effect is not as apparent in the last few runs since any material showing

TABLE 23

Evaporator Operating Results

RUN	FEED SOLUTION				CONDENSATE		DECONTAMINATION FACTOR		FINAL CONCENTRATION FACTOR	REMARKS
	*p*H	Density	Gross (3d/m/ml)	Gallons	*p*H	Gross (3d/m/ml)	Cyclone Separator	Overall		
Test Run 1	8.7	1.038	3.69×10^5	6865	--	448	---	824	6.75:1	Synthetic Feed
Hot Run 1	10.6	1.007	5.74×10^5	19215	8.9	501	---	1146	28.5:1	W5 Waste
Hot Run 2	10.7	1.006	8.31×10^5	24095	8.9	1130	3.04	735	42.5:1	W5 Waste
Hot Run 3	10.4	1.023	3.7×10^5	30897	2.3	2.7×10^4	---	13 (1)	45:1	W5 Waste Evap.*p*H < 6
					8.2	649	3.41	570		Evap.*p*H > 8
Hot Run 4	9.2	1.007	4.11×10^5	20214	7.2	409	2.69	1005	52:1	W5 Waste
Hot Run 5	7.9	1.006	9.25×10^5	30346	6.9	294	1.94	3160	47.6:1	W5 Waste
Hot Run 6	10.9	1.016	8.99×10^5	11732	8.3	298	3.49	3020	15:1	Redox & W5 Waste
Hot Run 7	10.8	1.015	8.47×10^5	17087	8.4	167	2.94	5070	25.4:1	Redox & W5 Waste
Hot Run 9	8.7	1.015	6.71×10^5	18696	3.0	7380	---	91 (1)	30.9:1	W5 Waste Evap.*p*H < 6
					7.7	345	4.25	1950		Evap.*p*H > 8

Note: 1. Foaming was present except in the first portion of Runs 3 and 9 at an evaporator *p*H < 6.

2. The condensate activity is for the material discharged to the Settling Basin during periods of foam free operation. About 10% of the condensate had to be recycled to W5 because of high activity from foamovers.

3. Run 8 was terminated in the early stages to perform maintenance work in the cell.

TABLE 24

Radiochemical Analysis of Feed and Condensate Solutions

		PERCENT OF TOTAL ACTIVITY							GROSS ACTIVITY (d/m/ml)	
Run	Solution	Ce	Cs	I	Ru	Sr	TRE-Ce	Activity Accounted For (Percent)	β	γ
4	Feed	34.0	4.5	22.5	3.7	8.1	10.9	84.7	4.11×10^5	921
	Condensate	*	7.39	70.4	4.04	8.79	4.75	95.4	409	0
6	Feed	9.32	18.0	2.81	21.8	2.02	2.52	56.5	8.99×10^5	2.37×10^3
	Condensate	*	*	0.9	20.2	1.5	2.07	24.7	298	0
7	Feed	*	18.3	1.02	17.1	0.9	1.21	38.5	8.47×10^5	2.62×10^3
	Condensate	*	*	7.29	24.4	1.31	1.85	34.9	167	0
9	Condensate (1)	*	3.57	64.3	25.7	.71	3.57	97.8	7580	*
	Condensate (2)	*	45.1	16.7	11.4	5.16	1.80	80.1	345	0

Notes: (1) Condensate from first part of run in which evaporator heel was acid.

(2) Condensate from second part of run in which evaporator heel was basic.

* Analysis not made.

TABLE 25

Ionic Analyses of Product Solution

(Composite Sample of Runs 6 & 7 Concentrate)

ANALYSIS	CONCENTRATION (gms/liter)		
	Sludge	Clear Supernate	Mixed Sample
U	8.0	0.2	1.1
Na	154.1	17.9	18.3
Ca	1.5	0.01	0.03
Mg	0.12	< 0.005	< 0.005
Fe	0.27	0.018	0.022
Al	6.5	7.5	7.8
Cl	0.008	0.006	0.009
CO_3	122.6	33.5	33.0
SO_4	10.7	0.52	0.54
NO_3	351.6	486.7	355.7
F	0.46	0.035	0.16
Total Solids	534.4	536.9	566.4

Sample specific gravity = 1.354

Gross $\beta = 1.8 \times 10^7$ d/m/ml

Gross $\gamma = 5.3 \times 10^4$ d/m/ml

pH = > 11

No percentage solids by volume or weight were determined. Due to extreme feed composition variations, these data are not representative of the composition of an average evaporator concentrate.

a trace of carry-over was returned to W5 without sampling. About 10% of the feed material had to be recycled to W5 because of excessive contamination by foaming.

A limited amount of experimental work during Run 2 with available antifoam reagents showed that 100 ppm of Dow Corning Silicon Antifoam "A" would suppress foaming for twenty-eight hours, 200 ppm of triphenyl phosphate was effective for twenty-one hours. Sulphonated castor oil, in any concentration, was an excellent foam promoter.

The other method of combatting foam is to operate with reduced liquid level so that part of the coils are exposed to act as a heated foam breaker. Of these two methods, the latter is the most effective. The current operating procedure is to add 100 ppm of Dow Corning Antifoam "A" every twenty-four hours and to keep some of the evaporator coils exposed at all times. This procedure is not infallible as foam-overs still occur occasionally. When this happens, the liquid level is further reduced, and more antifoam reagent is added.

The feed solutions contain finely divided solids and collodial material, dissolved carbon dioxide, salts known to have foaming tendencies, and may contain small amounts of detergents from decontamination operations. In addition the *p*H is quite high and the feeds are evaporated to high salt concentrations. These conditions all contribute to foaming, making it difficult to eliminate. The influence of *p*H on foaming is probably the largest single factor as shown by the first part of Runs 3 and 9 in which acid was added to the evaporator at the start of each run. There was absolutely no sign of foaming until the acid was neutralized by the basic feed and the evaporator *p*H become > 6, at which time foaming occurred. The routine addition of acid to the evaporator to maintain a *p*H < 6 is impractical because of the large quantities of acid required and the accelerated evaporator corrosion. Also, under acid conditions almost complete distillation of iodine into the condensate occurs, offsetting the gain in decontamination factor which results from foam elimination. No marked reduction in foaming was observed in Runs 4 and 5 in which the feed was slightly less basic than usual.

The problem of foam elimination should be investigated further when a safe margin is gained in the underground waste storage tanks. Suggested devices include mechanical and heated foam breakers in the tank vapor space, packed or bubble cap towers, and partial condensers or acidified cold water sprays in the vapor line to agglomerate active particles so they are more effectively removed by the cyclone separator.

In spite of foam, evaporation rates of ~ 300 gallons per hour have been maintained.

Condensate Monitoring. Development work on monitoring the condensate steam during the construction period showed that very efficient plating of active material onto the surface of the immersed Geiger tube and stainless steel occurred. The condensate monitor was redesigned so the Geiger tube was suspended horizontally a short distance above a free flowing liquid surface. A shield around the tube isolates it from any immersed metal surfaces so only the radiation from the liquid is received. This instrument is useful for detection of sudden increases of activity from a foam-over but has not been successfully calibrated in terms of counts per milliter of condensate because of background and other effects resulting from foaming. Refinement of design should eliminate these undesirable features.

Evaporator Costs. The cost of construction for the chemical waste evaporator is summarized below.

ORNL COSTS

Material	$18,394.	
Engineering	9,662.	
Construction Labor	4,285.	
Overhead	13,240.	
	$45,581.	

J.A. JONES COSTS

Labor and Material	44,000.	
	$89,581.	Total

The operating cost of the evaporator, excluding depreciation of the equipment and analytical costs, is estimated to be as follows for the first nine runs assuming a rate of 300 gallons per hour (approximately 54,000 pounds of water per day evaporated):

Steam	62,400 lb/day	$92.60
Cooling water	144,000 gal/day	11.50
Antifoam "A"	1 lb/day	7.00
Maintenance of Instruments and Equipment		8.00
Miscellaneous Reagents		3.00
One operator, 24 hour coverage, @$4.00/hr. (including overhead)		96.00
		$218.10

Cost/gallon of waste evaporated = $0.0303

If we assume that the life of the evaporator will be three years, and amortize the initial investment accordingly, the cost of evaporation per gallon, assuming an evaporation rate of 300 gallons/hour, for 325 days per year is:

Daily operating cost	\$218.10 per day
Amortized equipment cost	91.80 per day
	\$309.90 Total
Cost per gallon evaporated	\$0.043 per gallon

Effectiveness of Evaporator in Reducing Active Waste Discharge to White Oak Creek. Very limited data have been accumulated to date (August 2, 1949) concerning the effectiveness of the evaporator in reducing the curie discharge from ORNL to White Oak Creek. In order to provide a yardstick with which to measure the evaporator performance with respect to the overall liquid waste disposal problem, data from July 24 to July 30, 1949, are presented.

During this period of operation the evaporator feed was supernatant solution from the metal waste storage tanks in which uranium has been precipitated and settled by the addition of sodium hydroxide. The ionic analysis of this waste supernate follows:

	mg/ml
Na	54.2
NO_3^-	15.9
$CO_3^{=}$	29.3
$PO_4^{\equiv}$	6.0
$SO_4^{=}$	132.0

The gross β and γ activity of this solution is as follows:

Gross β: 2.6×10^6 c/m/ml

Gross γ: 0.58×10^3 c/m/ml

The solution $pH = 10\text{-}11$, and the specific gravity is 1.1.

Operation of the evaporator for the week of July 23 - July 30:

No. of runs	10
Total feed volume	68,673 gallons
Total concentrate volume	7,565 gallons
Concentration factor	9:1
Total curies in feed	151.5 curies
Total curies in condensate	0.179 curies
Decontamination factor (overall)	850

It should be noted here that the evaporator was operated at the rate of 600 gallons per hour during this period in order to free much needed waste storage capacity in the tank farm. When the tank farm supernates have been evaporated to the point where there is no longer a serious shortage of storage space, the evaporation rate, and hence the activity carry-over will be reduced.

DECONTAMINATION OF ORNL WASTES BY CRYSTALLIZATION

No chemical development work has been done on this problem during this quarter because more effort has been concentrated on RaLa development. However, equipment is being assembled for semi-works scale crystallization of ORNL wastes. The equipment consists of a 25 gallon evaporator and crystallizer, a fractioning column for decontamination and separation of water and nitric acid, and a 10 inch bowl American Tool and Machine Company centrifuge for separating and washing crystals. Plans are to treat concentrate from the ORNL evaporator, Hanford type wastes, TBP wastes, UAP wastes, and $Al(NO_3)_3$ wastes in this equipment with the object of decontaminating the inactive salts sufficiently to be discarded, thus saving storage space and concentrating the fission activities in a small volume readily available to the isotope production group.

RaLa PROCESS DEVELOPMENT

SEMI-WORKS

The present process involves precipitating barium sulphate from the nitric acid solution of an irradiated uranium using lead as a carrier. The sulphate precipitates are separated from the slug solution by decantation, metathesized to carbonates, dissolved in nitric acid and electrolyzed to plate out the lead carrier. The product is purified by precipitation from nitric acid and HCl-ether over a sintered glass filter. Waste losses have varied widely but have always been high, attributable principally to the mechanical inefficiency of the decantation operation. Centrifugation and filtration were proposed as alternatives and half-scale equipment was installed for testing and to determine what process variations were advisable. Investigatory work has been in accord with the following outline:

1. Sulphate separation step
   - A. By filtration,
   - B. By centrifugation,
     - a. with lead carrier to aid precipitation
     - b. without lead carrier;
2. Methathesis (conversion of sulphate precipitate to carbonate) step
   - A. Using filtration,
   - B. Using centrifuge
     - a. metathesizing both lead and barium with carbonate solution
     - b. metathesizing barium and dissolving the lead with caustic-carbonate solution to effect a rough lead removal.

A full-scale, resin absorption column, has been erected to which the above metathesis product is fed to determine the effectiveness of resin absorption in refining the barium, in place of the purification steps now in use.

A brief outline of the proposed process is as follows:

1. Nitric acid slug dissolution.
2. Sulphate precipitation (using lead carrier).
3. Sulphate separation.
4. Metathesis, re-solution, make 0.5 *M* in Ac^-, adsorption on Doyex 50 resin column.
5. Caustic elution for removal of lead.

6. pH^3 citrate elution for removal of iron, chromium, nickel.
7. pH^6 citrate elution for removal of strontium.
8. 6 *N* HNO_3 elution of barium product.
9. Evaporation of product to dryness for volume reduction, and re-solution.

More work will be necessary before conclusive results are obtained, however the results below have been obtained:

	BARIUM LOSSES
SULPHATE SEPARATION	
Filtration (micro-metallic G)	0.1-0.5%
Centrifugation	
with carrier	0.5%
w/o carrier	22%
METATHESIS	
Filtration	0.1-0.2%
Centrifugation	
metathesis only	0.3%
metathesis and Pb removal	1.5- 3%
RESIN ABSORPTION	
Barium Yield	81%
Barium Loss	1%
Material Balance	82%
OVER ALL MATERIAL BALANCE	87%

These results indicate that both filtration and centrifugation are applicable. On the basis of ease of installation a vacuum filter was recommended.

Experimental work will continue to give further information on filter operation, and to refine the resin absorption process. The latter ultimately will be operated on RaLa plant wastes to demonstrate its ability to function under high radiation intensities.

PURIFICATION OF Ba^{140} BY ION EXCHANGE

Ion exchange is being considered for use in the RaLa process as it offers the advantages of ease of remote control and higher purity of product. Two ion exchange processes have been developed for the purification and recovery of Ba^{140}. These new alternate processes may be added subsequent to either the lead sulfate precipitation step or the metathesis step, and will replace the metathesis, electrolysis, and ether hydrochloride steps.

In the Acetate-Citrate Process, the barium-lead sulfate precipitate is dissolved in 1 *M* sodium acetate or the acid metathesis product is made 0.5 *M* in sodium acetate and passed through a Dowex 50 resin column where barium, strontium, lead, and sodium are absorbed while most of the iron and chromium pass through as acetate complexes. The lead, iron, strontium, and sodium are selectively eluted and the pure barium finally eluted in 6 *N* HNO_3 with a yield of approximately 99%. In the Alkali-Citrate Process, the barium-lead sulfate is dissolved in 6 *M* NaOH ($CO_3^{=}$ free) and diluted to 0.6 *M* NaOH. Alternately the acid metathesis product may be made 0.5 *M* in NaOH, centrifuged to remove the hydrous oxides of iron, chromium, and nickel and passed through the resin bed. In both cases the lead passes through as the plumbite and barium is absorbed, freed from strontium, and recovered as above. These processes have been verified in the laboratory on 1/100 scale and are now in semi-works trial on full scale (see flowsheet Fig. 3).

The elution curves of barium with 6 *N* HNO_3, 6 *N* HCl or 2 *N* HNO_3 show the 6 molar nitric acid to be about eight times as efficient as the HCl due to the complexing action of the nitrate. Therefore, 6 *N* HCl can be used to selectively elute the sodium from a column with a negligible barium loss.

If the maximum amounts of iron, nickel, and chromium expected to be present in the RaLa process are precipitated as the hydroxide in the Alkali-Citrate Process, they will carry 2-5% of the barium. However, the use of ammonium nitrate, *p*H 5, as a washing agent will reduce the loss to less than 1%.

Preliminary laboratory experiments have shown that approximately 99% of the impurities, iron, chromium, nickel, and possibly lead, can be separated from barium by adsorption on the acetate form of IR4B or Dowex A2 anion resin from a 0.5 *M* sodium acetate solution. These results are being rechecked.

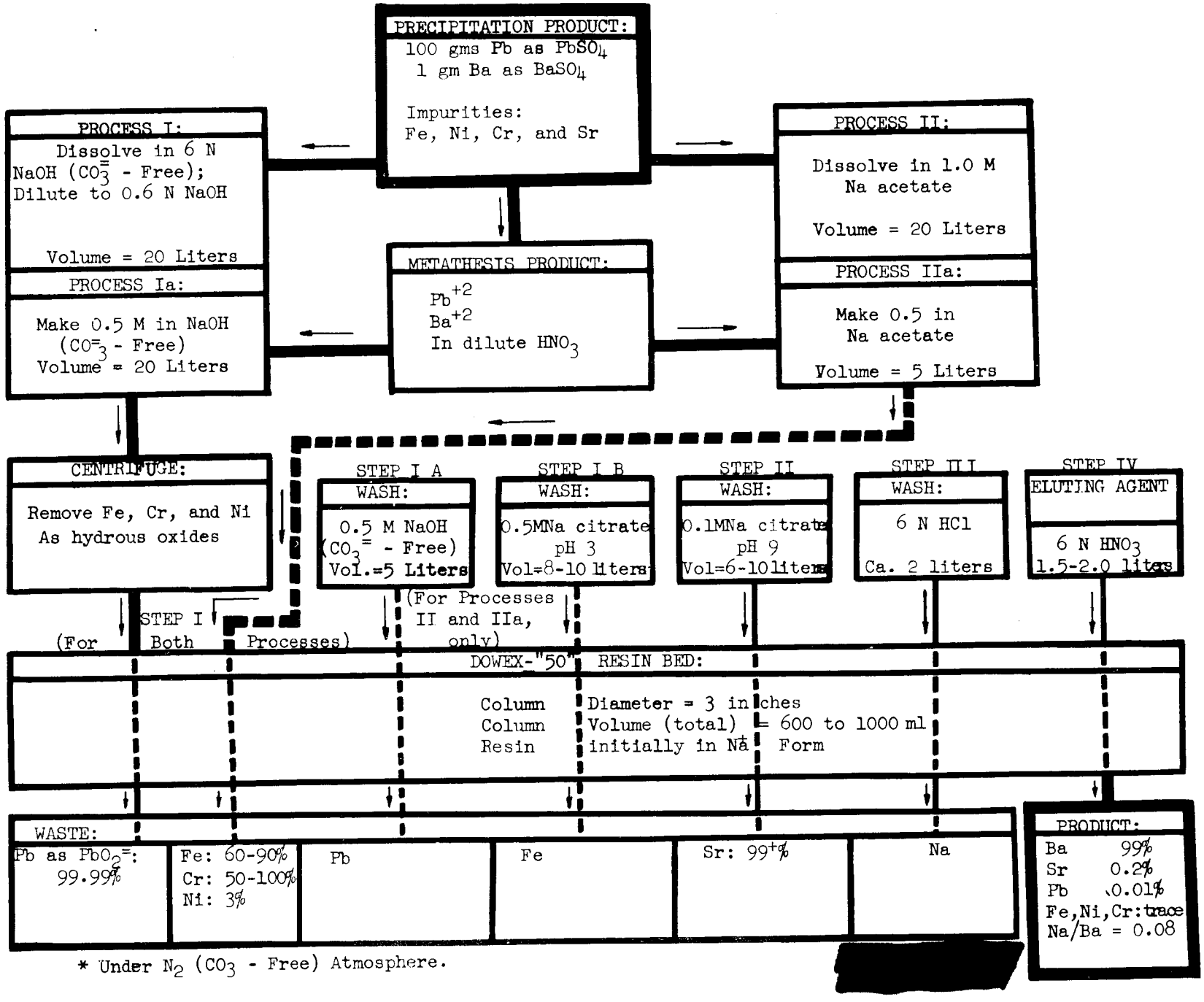
An attempt is being made to develop an up-flow resin column so that if gas is formed as a result of water decomposition from radiation, the bubbles

[redacted]

FIGURE 3

FLOWSHEET: PROPOSED RALA ION EXCHANGE PROCESSES

Drawing # 7536



* Under N_2 (CO_3 - Free) Atmosphere.

[redacted]

will escape through the expanded end and thus not block the column. After an investigation of bed expansion as a function of particle size and flow rate, a resin column was prepared containing known percentages of particles ranging from 40-240 mesh. For a flow rate of 0.56 cc/cm^2/min, the bed expansion was 25%. During operation, each particle assumes a position relative to its size so that the bed does not "churn". An encouraging separation of strontium and barium has been made with this column.

The use of IRC-50 resin is being investigated since the volume of acid needed for barium elution would be considerably less, thus reducing evaporation volume. Two disadvantages are: (1) barium and strontium are held much more strongly on IRC-50 than Dowex 50 and thus a much larger volume of citrate elutriate is needed. Also, the degree of separation of barium and strontium is poor so that a new acid elution process will have to be developed; (2) since H^+ ion replaces all other cations, HCl cannot be used to separate sodium and barium. However, the change in capacity of the resin for sodium with *p*H has been determined so that by washing the resin with a buffer solution of sodium acetate at a *p*H of about 4, it is expected that the bulk of the sodium will be eluted leaving the Ba in the resin for subsequent removal with acid.

25 PROCESS DEVELOPMENT

PILOT PLANT

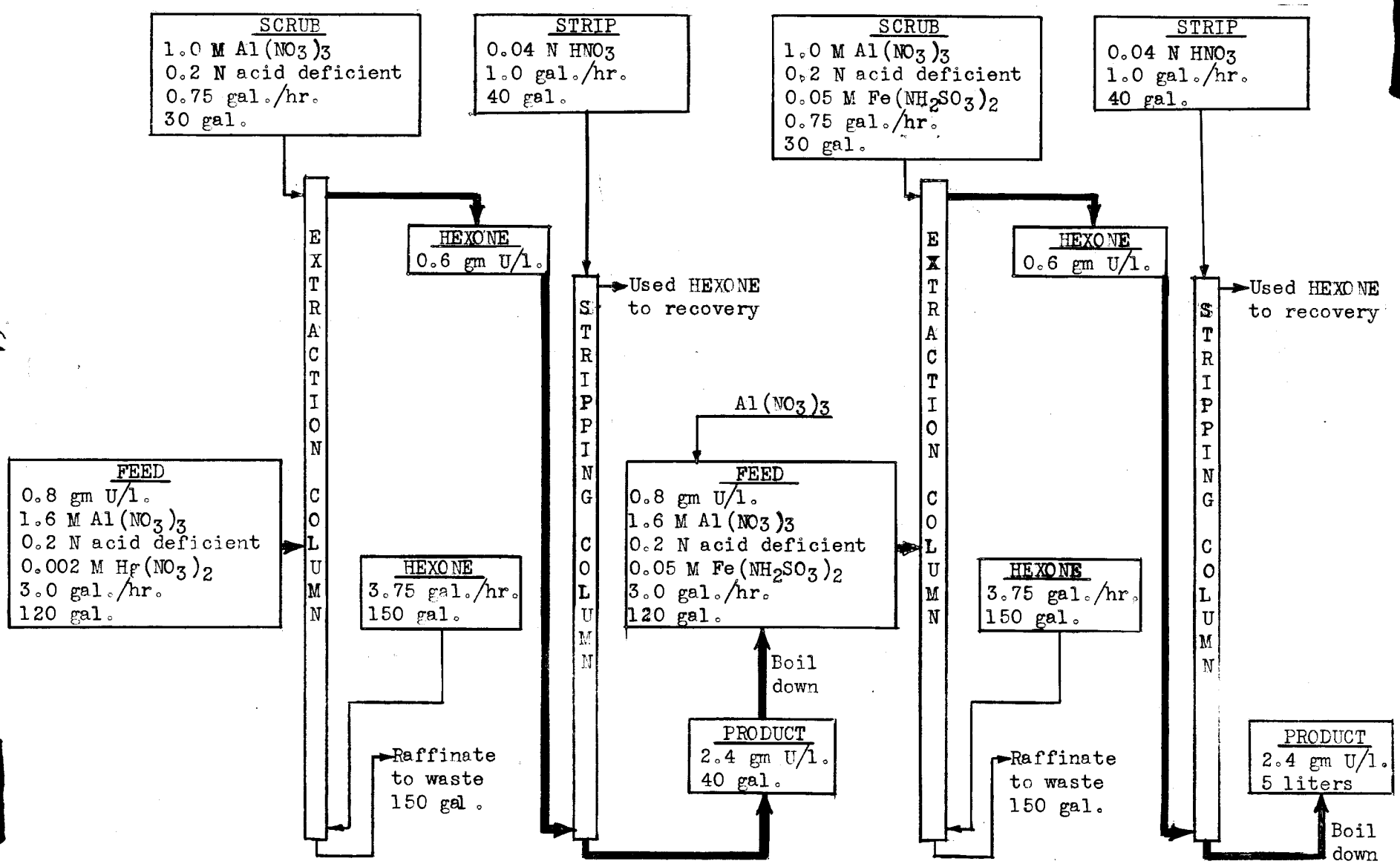
In August, 1948, the ORNL Pilot Plant phase of the 25 process investigation was interrupted in favor of the Redox investigation. Upon completion of the Redox program, the 25 process development program was resumed, and ten two-cycle 25 recovery runs using Hanford irradiated uranium have been completed. The primary objectives of these runs were to test the 25 process for mechanical operability in equipment which had been modified for the Redox investigation, to check uranium balances at the low uranium concentrations (1 g/*l* and below) of the 25 system, to establish minor process modifications suggested by the Redox investigation, and to make minor flowsheet charges suggested by recent process developments in the Laboratory and Semi-Works.

The two-cycle beta decontamination factors obtained for the 25 runs were consistently about 3×10^5, corresponding to a gamma decontamination of about 1×10^5. Perhaps of more significance was the fact that the gross beta activity in the uranium product stream was less than 40 counts/min/ml, one-third of the beta activity of natural uranium. Higher decontamination factors may be expected when the initial activity is high enough to obtain a better counting range for the second cycle product. The apparent uranium losses on the first runs were as high as a few per cent, but these losses were traced to uranium remaining in the system after the Redox runs, and to certain operational procedures used for column startup and shutdown. The average two-cycle column loss for the last four runs was 0.02%, and the average filtration loss was 0.15%.

The 25 process flowsheet in effect at the end of the runs discussed above is shown in Fig. 4. The only significant flowsheet change made during the past period was the elimination of ferrous sulfamate in the first cycle because of the formation of insoluble mercuric sulfamate. Mercuric nitrate is required to catalyze the dissolving of the aluminum-uranium alloy slugs.

At the conclusion of the runs described above, the entire pilot plant system was decontaminated in order that equipment in the cells could be inspected, repaired, and cleansed of all natural uranium. During the next quarter the pilot plant phase of the 25 process investigation will terminate with final verification runs, which will consist of the processing of 2200 grams of enriched (95%) U^{235}. This material is in the form of an alloy of 4% uranium and 96%

Figure 4



"25" PROCESS PILOT PLANT FLOWSHEET
8-15-49

FIGURE

aluminum, the approximate composition of the alloy to be used in the fuel assemblies of the Materials Testing Reactor. This material, consisting of 217 4-inch Hanford type slugs, has been irradiated at Hanford to 5% depletion of the U^{235}.

ALUMINUM-URANIUM ALLOY SLUG DISSOLUTION

The development of a procedure is nearing completion for dissolving aluminum alloy slugs containing 4% U^{235}. The process recommended from Semi-Works development comprises gradual addition of concentrated nitric acid to the slugs which are immersed in 13 liters of water containing 20 g of mercuric nitrate catalyst per kilogram of slug weight. Over a 14 hour period, 8.5 liters of 70% HNO_3 are added per kilogram of slug, and the temperature is increased progressively from 90° C to 102° C. After 10 hours additional digestion at 102-104° C, 95-100% of the slug weight is dissolved and the final solution is 1.7-1.8 *M* in $Al(NO_3)_3$ and 1.0-0.2 *N* in excess HNO_3. Further digestion may be employed if necessary (Table 26).

Problems of major concern in development of the dissolving process were:

(1) Controlling the Initial Dissolving Rate to Avoid Uncontrolled Reaction Which Could Be Disastrous. There has been no tendency toward uncontrolled or spasmodic dissolving under the recommended conditions. Excess acid does not accumulate during the early stages of dissolving, and violent reaction appears impossible later.

(2) Obtaining Complete Dissolution of Slugs With a Final $Al(NO_3)_3$ Concentration over 1.5 M. Most of the restrictions placed on the process are caused by the requirement of 95% or more complete dissolving. It is now well established that (a) 1.8 *N* is the maximum attainable final solution concentration, (b) mercuric nitrate catalyst equivalent of 1.5% of the slug weight is the minimum quantity, and (c) 0.5-1.0 *N* is the optimum excess HNO_3 concentration during dissolving.

The progress of dissolving was followed by measuring the specific gravity and *p*H of the solution. These data, with the known amount of acid added, enabled a quick determination of the amount of aluminum dissolved while dissolving was in progress. The temperature rise in the cooling water passing through the dissolver jacket furnished a convenient index of reaction rate, particularly during the early stages of dissolving.

TABLE 26

Aluminum-Uranium Alloy Slug Dissolution, Semi-Works Runs

Nitric acid (70%) added over 6-15 hour periods to dissolver containing 1.36 in. diameter cylinder Al slugs (some contain 4% uranium) 1 in. to 8 in. in length, and immersed in water heel containing mercuric nitrate catalyst as indicated, expressed as percent by weight of slug temperature controlled by water-steam jacket.

NUMBER	NUMBER RUNS AVERAGED	PERCENT CATALYST	TYPE SLUG	NUMBER OF SLUGS	TYPE OF DISSOLVER	Ml OF WATER HEEL PER gm SLUG	Ml 70% HNO_3 PER gm SLUG	DISSOLVING TEMPERATURE °C			DURATION OF RUN-HOURS	FINAL CONDITIONS			
								First Quarter	Second Quarter	Last Half		$Al(NO_3)_3$ Molarity	Acid Normal	Percent of Slug Weight Dissolved	
1	1	2.0	Al-4	3	S/S	12.8	8.5	99	104	105	22	1.78	0.1	100	DEMONSTRATION SERIES
2	1	2.0	Al-4	3	S/S	13.0	8.1	98	100	104	22	1.65	0.5	97	
3	1	2.0	Al-4	3	S/S	12.8	8.3	93	103	105	22	1.68	0.8	97	
4	1	2.0	Al-4	2	S/S	13.2	8.2	95	101	101	11	1.63	0.9	100	
5	1	2.0	Al-4	2	S/S	12.8	8.5	97	99	103	14	1.56	1.2	93	
6	1	2.0	Al-4	1	S/S	13.6	8.3	93	100	104	22	1.65	0.5	100	
7	1	2.0	Al-4	1	S/S	12.4	8.2	98	100	103	23	1.59	1.3	97	
8	1	1.5	Al-4	2	S/S	13.1	8.4	92	103	103	21	1.64	0.7	100	
9	1	1.5	Al-4	2	S/S	13.2	8.5	90	103	103	40	1.66	1.0	95	
10	1	2.0	Al-4	1	S/S	11.4	8.3	96	101	103	27	1.45	2.7	74	EXPERIMENTAL SERIES
11	1	2.0	*Al-4	1	S/S	13.0	8.9	102	104	105	52	1.70	0.7	100	
12	1	1.0	*Al-4	1	S/S	10.3	9.4	87	101	102	6	1.83	0.3	100	
13	1	1.0	*Al-4	1	S/S	9.4	9.4	87	103	103	10	1.26	3.6	71	
14	4	2.0	Al	1	S/S	10.3	9.4	87	101	102	7	1.9	0.5	99	
15	1	1.0	Al	1	S/S	10.3	9.4	95	95	95	6	1.4	2.0	72	
16	5	1.0	Al	1	S/S	9.8	9.4	86	101	102	9	1.7	1.8	85	
17	3	0.6	Al	1	S/S	10.6	8.5	83	97	101	9	1.1	2.6	42	
18	1	1.0	Al	-	Glass	10.7	6.8	Approx. 100			6	2.1	-1.6	100	
19	2	0.6	Al	-	Glass	10.6	9.8	75	93	97	11	1.3	3.0	71	
20	2	0.4	Al	-	Glass	10.7	8.2	65	75	90	10	1.2	2.6	52	
21	1	0.1	Al	-	Glass	11.1	7.3	70	70	77	8	0.3	5.4	17	

The dissolving characteristics of the Al-4% U alloy slugs used for final Semi-Works demonstration runs were only slightly different from the behavior of pure aluminum slugs used in other investigations. The alloy slugs seemed to dissolve more smoothly, with less tendency to stop dissolving in acid solutions than the pure aluminum, but the difference was not great.

No precipitate, or crud, was observed in the final solution from unjacketed alloy or pure Al slugs, even after addition of ammonia to produce acid-deficient solutions. The crud observed in solutions from jacketed slugs must come from bonding material under the jacket. The crud capacity of a sintered stainless steel filter plate of 10 micron "G" porosity is being determined to guide pilot plant operations.

23 PROCESS DEVELOPMENT

THORIUM RECOVERY

An extraction process using tributyl phosphate as the solvent for the recovery and decontamination of thorium from 23 raffinates has been developed. Results of countercurrent batch extraction runs indicate that a thorium recovery of greater than 99.9% with a beta decontamination factor of 1.7×10^3 is possible.

The extraction of thorium with tributyl phosphate has been shown to be a rapidly reversible reaction with the optimum total nitrate concentration at 2-6 *M*, and the reaction compound to be three mols of tributyl phosphate per mol of thorium. Under these conditions protactinium was not extracted; however, it was found that tributyl phosphate extracted protactinium from 11 molar nitric acid feeds to the extent of about 7% per equal volume pass.

Batch scrub data indicate that a beta decontamination factor of 50 is obtained with a 4-6 *M* HNO_3 scrub of the thorium-tributyl phosphate complex.

Seven organic solvents, butyl analine, butyl sulfone, butyl benzene, butyl propionate, butyl oxalate, butyl urea, and butyl nitrate have been scouted for thorium extraction with none showing promise.

A total of 256.6 mg of 23 from one four-inch Hanford bombarded (cooled two years) thorium slug has been extracted and concentrated to 50 ml using dissopropyl ether as the extracting solvent. This separation was made to obtain actual 23 raffinates to be used in studying thorium recovery.

SEMI-WORKS DESIGN AND CONSTRUCTION

The construction of the 1300 Area Semi-Works for the demonstration of a solvent extraction process for the recovery of U^{233} from irradiated thorium is now approximately 90% complete. Pressure testing of lines, tanks, and other processing equipment is in progress. Preliminary operational tests and dummy runs for training operating personnel will be made during the coming period.

TABLE 27

Effect of Nitrate Ion on Extraction and Scrubbing of Thorium

EXTRACTION FEED: 0.06 *M* $Th(NO_3)_4$, 0.01 NaF, 0 to 6 *M* HNO_3

EXTRACTANT: 50% Tributyl phosphate - 50% hexane (equal volume pass)

SCRUBBING ORGANIC: 50% Tributyl phosphate - 50% hexane, 0.056 *M* Th

AQUEOUS: 0 to 6 *M* HNO_3

DISTRIBUTION COEFFICIENT	MOLARITY OF NITRIC ACID IN FEED AND SCRUB				
	0	1	2	4	6
Ext'n (organic/aqueous)	0.021	0.8	1.85	4.4	6.4
Scrub (aqueous/organic)	5	1.1	0.53	0.24	0.16

DRY FLUORIDE DEVELOPMENT

Two runs have been completed in which uranium hexafluoride was prepared by direct fluorination of uranium metal. In the first run, 12.5 grams of metal were completely fluorinated, and in the second run 46.5 grams of metal was charged and 14.5 grams were reacted. At a temperature above 300° C the reaction was vigorous and could be controlled only by regulating the fluorine flow rate. Little or no reaction was noted at less than 300° C. Heat transfer into the cooling water was hindered because most of the reaction was limited to the small area where the fluorine first came in contact with the metal, and was so poor that the uranium metal fused when it was fluorinated at a rate of 0.8 grams per minute. At this rate, 1700 calories are liberated per minute. When the fluorine flow rate was reduced, there was indication that the heat transfer was sufficient to remove the heat of reaction; however, the reaction rate could not be estimated since the flow-meter employed would not detect such low fluorine flow rates. A considerable amount of greenish-yellow powder, probably UF_4, was noted on the walls of the reactor. If this is uranium tetrafluoride, it may be corrected to uranium hexafluoride by heating the reactor and discharge lines and passing fluorine through them.

In the next period, a flowmeter with the range of 20-200 ml/min will be installed. This amount of fluorine will react with 0.06-0.6 grams of uranium per minute and liberate 130-1300 calories of heat per minute. The series of runs to determine if the reaction can be controlled and to determine the limitations of this Laboratory reactor will be continued.

ZIRCONIUM-HAFNIUM SEPARATION

The possibility of separating zirconium from hafnium by solvent extraction with tributyl phosphate has been investigated. Preliminary work indicates that a separation factor of 17 may be obtained, with zirconium extracting preferentially.

EQUIPMENT DEVELOPMENT

BELLOWS PUMP TEST

Two Argonne bellows pumps (ref. drawing No. CS-1015, Argonne National Laboratory) which will be used to pump "hot" aqueous feed to the extraction columns in the Semi-Works 1300 operation have been pre-tested and calibrated one for water and one for $Th(NO_3)_4$ solution. No operational difficulties were encountered.

The calibration curves were prepared from, in each case, 13 or more determinations of rotameter reading and measured volumetric flow rate over the range of bellows settings. Maximum flow rates were 130 and 135 ml/min with water, and for the latter pump 118 ml/min with $Th(NO_3)_4$. The measured volumetric flow rate correlated more closely with the bellows setting than with the rotameter reading, probably because of the effect of the pulsating flow on the rotameter reading. The reproducibility of measured flow rate at a given bellows setting was found to be 3% or less (with water).

The method of connecting the air chamber "surge pots," provided to dampen pulsation, was changed so that the pump discharged passed directly through the surge pot, and a valve in the discharge line was partially closed. Before these changes were made, the rotameter float would pulse 7 rotameter scale readings, for example, and the pulsation was reduced to 0.5 scale readings afterward.

Both pumps were operated by a single electric motor (¼ hp) through a gear reducer at a speed of 45 positive bellows strokes per minute. Each pump delivered the test solutions to a height of 15 feet through a ½ in. saran line. Rotameter readings were noted at each setting of the bellows flex control (micrometer barrel graduated from 0 to 42 turns) and discharge rates were obtained by catching the test solution in a graduated cylinder for a period of 10 minutes.

PROTECTIVE COATINGS FOR RADIOACTIVE DECONTAMINABLE SURFACES

The use of protective paints, plastics and floor materials as a means of controlling contamination in Radiochemical Laboratories and attendant facilities has been investigated in a joint program conducted by the Technical and Biology

Divisions. The Biology Division determined the susceptibility of these materials to contamination and their subsequent ease of decontamination. The Technical Division determined the chemical resistance of these materials to common laboratory reagents.

These data have been combined and are presented in five tables:

Table 28 Non-Strippable Coatings

Table 29 Strippable Coatings

Table 30 Baked Coatings

Table 31 Miscellaneous Materials (Flooring, Plastic Sheets, Tapes)

Table 32 Structural Materials (Steel, Lead, Concrete, Glass, etc.)

For convenience, and since chemical resistance properties and decontamination properties are not directly comparable, the materials in each table (with the exception of Table 32) are presented in a decreasing order of usefulness based on chemical resistance tests. Table 32 presents Spill Indices* for commonly used laboratory structural materials and is a means of evaluating Spill Indices in Tables 28-31

Although abrasion and cost data are not known, the above tables are presented as a guide to aid the potential user in selecting coatings or materials best suited for his particular case.

Conclusions. It was concluded that many coatings are useful in Radiochemical Facilities and that several could probably replace stainless steel under favorable circumstances.

Introduction to Protective Coatings Tables.

(a) 3/8 in. × 5 in. dowels (aluminum and wood) coated dried according to the manufacturers specifications.

(b) Immersion in 3 *M* HNO_3, HCl, H_2SO_4 and NaOH for 7 days. Condition of sample checked every two hours.

(c) Reagent temperature—20-25° C.

METHOD OF RATING	METHOD OF JUDGING FAILURE
E-Excellent - No attack in 168 hours (1 week).	1. Pronounced discoloration of reagent or test rod.
G-Good - Failure in 120-167 hours.	2. Blistering or cracking.
F-Fair - Failure in 48-119 hours.	3. Tacky or slimy surface.
P-Poor - Failure in less than 48 hours.	4. Appearance of pin holes.

* The higher the spill index, the greater the ease of decontamination.

Decontamination and Contamination Susceptibility Tests.
(See Tompkins, P. C., Bizzell, O. M., "Radiation Decontamination of Laboratory Surfaces", ORNL 381 - In press)

METHOD OF RATING

$$\text{Spill Index (S.I.)} = -\log \frac{\text{Activity after cleaning}}{\text{Activity applied}}$$

The activity level of the D.I.'s air dried placques was approximately 100 times greater than that of the susceptibility (rinsed) placques.

$$\text{Susceptibility (\% Adsorbed)} = \frac{\text{Activity on surface before cleaning}}{\text{Activity applied}} \times 100$$

$$\text{Decontamination Index (D.I.'s)} = \log \frac{\text{Activity on surface before cleaning}}{\text{Activity on surface after cleaning}}$$ and equal to Step I + Step II.

Step I = Reagent decontamination by rinsing.

Step II = Reagent decontamination by scrubbing.

$\frac{\text{Activity Level}}{\text{Counter}}$ = 1.2×10^7 c/min read on the third shelf of a bell type Geiger Muller Counter.

Isotopes - Carrier free (i.e., none added during tests) P^{32}, Ba^{140} I^{131} as produced by ORNL.

TABLES 28-32

Protective Coatings for Surfaces Exposed to Radioactivity--

Chemical Stability and Decontamination Data

TABLE 28

Non-Strippable Coatings

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	* Gross Avg. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I	Step 1 D.I.	Step 2 D.I.	
Devoe and Raynolds Co., Inc. Louisville 1, Ky.	Devan Resin Solution K-5925	Clear	E	E	E	E	G	4	P^{32}	4.1	0.2	3.4	2.8	0.6	
									Ba^{140}	5.5	0.02	3.8	3.5	0.3	
									I^{131}	2.6	0.2	1.9	1.8	0.1	
U.S. Stoneware Akron 9, Ohio	Duralon - 35	Black	E	G	E	E	E	5	P^{32}	5.7	0.05	4.4	3.8	0.6	Can be applied only to porous media such as concrete or wood. Curing time 2 weeks.
									Ba^{140}	4.0	0.7	4.3	3.5	0.8	
									I^{131}	4.0	0.1	3.0	2.1	0.9	
American Pipe and Const. Co. Los Angeles 54, California	Amercoat - 31	Red	E	E	E	E	P	5	P^{32}	6.4	0.05	5.1	3.4	1.7	
									Ba^{140}	6.0	0.03	4.5	4.0	0.5	
									I^{131}	3.3	0.02	1.6	1.6	0.02	
American Pipe and Const. Co. Los Angeles 54, California	Amercoat - 44	Black	E	E	E	E	P	6	P^{32}	7.5	0.01	5.5	3.0	2.5	
									Ba^{140}	6.8	0.01	4.8	3.6	1.2	
									I^{131}	3.0	0.02	1.3	1.3	0.03	
American Pipe and Const. Co. Los Angeles 54, California	Amercoat - 55	Black	E	E	E	E	P	6	P^{32}	7.3	0.01	5.3	3.2	2.1	
									Ba^{140}	7.2	0.01	5.2	4.1	1.1	
									I^{131}	3.3	0.01	1.3	1.2	0.1	

* For comparison of spill indices with standard materials such as stainless steel, lead and pyrex glass - See Table 32.

(Continued on next page)

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
Bisonite Co., Inc. Buffalo 1, New York	M-100; M-101	Gray	E	E	E	E	P	5	P^{32}	5.6	0.02	3.9	3.1	0.8	
									Ba^{140}	6.8	0.005	4.5	3.5	1.0	
									I^{131}	4.0	0.02	2.3	2.2	0.1	
Bisonite Co., Inc. Buffalo 1, New York	M-109	White	E	E	E	E	P	4	P^{32}	4.0	0.2	3.3	2.9	0.4	
									Ba^{140}	4.9	0.3	4.4	3.8	0.6	
									I^{131}	3.4	0.03	1.9	1.7	0.2	
Corrosite Corp. New York 17, NY	Plastic Coating	Gray	E	E	E	E	P	5	P^{32}	5.6	0.02	3.9	3.2	0.7	
									Ba^{140}	5.4	0.03	3.9	3.7	0.2	
									I^{131}	3.5	0.01	1.5	1.4	0.1	
Corrosite Corp. New York, 17, NY	Plastic Coating - 8228	Green	E	E	E	E	P	4	P^{32}	4.1	0.2	3.4	3.0	0.4	
									Ba^{140}	5.1	0.2	4.4	3.9	0.5	
									I^{131}	3.1	0.02	1.4	1.4	0.1	
The Garland Co. Cleveland, Ohio	Acanal	White	E	E	E	E	P	3	P^{32}	3.1	0.4	2.7	2.5	0.2	
									Ba^{140}	3.9	0.3	3.4	3.2	0.2	
									I^{131}	3.2	0.02	1.5	1.3	0.2	
The Glidden Co. Cleveland, Ohio	RL-8222 - Methacrylate	White	E	E	E	E	P	4	P^{32}	4.1	0.2	3.4	3.1	0.3	
									Ba^{140}	3.9	0.04	2.5	2.5	0.04	
									I^{131}	4.1	0.01	2.1	1.9	0.2	
The Glidden Co. Cleveland, Ohio	RL-8319 - Vinyl	White	E	E	E	E	P	4	P^{32}	5.1	0.02	3.4	2.6	0.8	
									Ba^{140}	4.0	0.03	2.5	2.4	0.1	
									I^{131}	3.8	0.01	1.8	1.7	0.1	
Gordon Lacey Chemical Products Company, Maspeth, L. I., N. Y. C.	J211E	White	E	E	E	E	P	4	P^{32}	4.4	0.04	3.0	2.7	0.3	
									Ba^{140}	5.3	0.02	3.6	3.1	0.5	
									I^{131}	3.2	0.008	1.1	1.1	0.03	
Gordon Lacey Chemical Products Company, Maspeth, L. I., N. Y. C.	A248B	Clear	E	E	E	E	P	6	P^{32}	7.1	0.007	4.9	3.5	1.4	
									Ba^{140}	6.4	0.02	4.7	4.0	0.7	
									I^{131}	3.7	0.003	1.2	1.2	0.03	

(Continued on next page)

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
Maurice A. Knight Company Akron 9, Ohio	Pyroflex Lacquer	White	E	E	E	E	P	5	P^{32}	6.3	0.02	4.6	3.4	1.2	
									Ba^{140}	5.5	0.06	4.3	2.6	1.7	
									I^{131}	4.1	0.005	1.8	1.7	0.1	
Maurice A. Knight Company Akron 9, Ohio	Pyroflex Lacquer	Gray	E	E	E	E	P	6	P^{32}	7.2	0.004	4.8	3.2	1.6	
									Ba^{140}	6.7	0.007	4.5	3.4	1.1	
									I^{131}	4.0	0.004	1.6	1.5	0.1	
Maurice A. Knight Company Akron 9, Ohio	Pyroflex Lacquer	Black	E	E	E	E	P	5	P^{32}	6.5	0.02	4.8	3.3	1.5	
									Ba^{140}	5.9	0.02	4.2	3.6	0.6	
									I^{131}	3.6	0.008	1.5	1.4	0.1	
Proxylin Products Inc. Chicago, Ill.	Proxcote 19-70-3	Clear	E	E	E	E	P	5	P^{32}	6.7	0.06	5.5	3.6	1.9	
									Ba^{140}	6.4	0.06	5.2	3.9	1.3	
									I^{131}	3.1	0.09	2.0	1.9	0.1	
Proxylin Products Inc. Chicago, Ill.	Proxcote 19-17-11	Clear	E	E	E	E	P	**	P^{32}	--	--	--	--	--	
									Ba^{140}						
									I^{131}						
United Chromium Inc. Detroit 7, Michigan	Ucilon 400-9	White	E	E	E	E	P	6	P^{32}	7.0	0.002	4.3	3.4	0.9	
									Ba^{140}	6.4	0.04	5.0	3.6	1.4	
									I^{131}	4.1	0.008	2.0	1.9	0.1	
Peninsular Chemical Products Company Van Dyke, Mich.	Penkote	Gray	E	E	E	E	P	1	P^{32}	0.1	3.7	0.6	0.6	0.03	Has been used on concrete floors. Excessive peeling and poor decontamination prohibits further use.
									Ba^{140}	1.4	0.2	0.7	0.7	0.04	
									I^{131}	1.0	0.3	0.5	0.5	0.02	
Midland Ind. Finishes Co. Waukegan, Ill.	L3X222 Silicone	Clear	E	E	E	E	P	7	P^{32}	7.1	0.006	4.8	3.3	1.5	
									Ba^{140}	8.2	0.004	4.2	3.6	0.6	
									I^{131}	5.6	0.008	1.5	1.4	0.1	
Lithgow Corp. Chicago 9, Ill.	LC-600 Gray	Gray	G	E	E	E	P	--	P^{32}	--	--	--	--	--	
									Ba^{140}						
									I^{131}						

** Coating brushed from plaque.

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
The Glidden Co. Cleveland, Ohio	RGL 19678 Rubber	White	P	E	E	E	P	5	P^{32}	5.9	0.01	3.9	2.9	1.0	
									Ba^{140}	4.7	0.03	3.2	2.9	0.3	
									I^{131}	3.2	0.02	1.5	1.4	0.1	
United Chromium, Inc. Detroit 7, Michigan	Ucilon 452	Gray	P	E	E	E	P	4	P^{32}	3.6	0.2	2.9	2.7	0.2	
									Ba^{140}	3.7	0.06	2.5	2.3	0.2	
									I^{131}	3.6	0.02	1.9	1.7	0.2	
Pratt & Lambert Buffalo 7, N. Y.	Vitralite	White	E	P	E	E	P	4	P^{32}	4.0	0.05	2.7	2.6	0.1	
									Ba^{140}	4.2	0.04	2.8	2.6	0.2	
									I^{131}	2.3	0.06	1.1	1.1	0.04	
Barrett Varnish Company, Cicero 50, Illinois	Varnish 20BK	Clear	E	P	E	E	P	6	P^{32}	8.0	0.005	5.7	3.3	2.4	
									Ba^{140}	7.1	0.01	5.1	4.1	1.0	
									I^{131}	3.3	0.008	1.2	1.1	0.1	
The Garland Co. Cleveland, Ohio	Supernite Varnish	Clear	E	P	E	E	P	5	P^{32}	5.9	0.01	3.9	2.5	1.4	Compatible with 815 concrete gray
									Ba^{140}	6.0	0.01	4.0	3.8	0.2	
									I^{131}	2.8	0.02	1.1	1.0	0.1	
The Glidden Co. Cleveland, Ohio	815 Concrete Gray	Gray	E	P	E	E	P	5	P^{32}	5.7	0.04	4.3	2.7	1.6	Excellent abrasion properties. Its use is planned in Isotope Production since no NaOH is used.
									Ba^{140}	6.2	0.02	4.5	4.0	0.5	
									I^{131}	3.4	0.01	1.4	1.2	0.2	
The Glidden Co. Cleveland, Ohio	No. 1 Black Coating 90-B-11	Black	E	P	E	E	P	4	P^{32}	5.8	0.03	4.3	3.6	0.7	
									Ba^{140}	4.8	0.07	3.6	3.4	0.2	
									I^{131}	2.3	0.08	1.2	1.1	0.1	
The Glidden Co. Cleveland, Ohio	RGL 18511 Chlorinated Rubber	Gray	E	P	E	E	P	4	P^{32}	4.0	0.5	3.7	3.2	0.5	
									Ba^{140}	5.4	0.01	3.4	3.1	0.3	
									I^{131}	3.2	0.02	1.4	1.3	0.1	

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
Maas and Waldstein Co. Newark, N. J.	Coprene 40106	Clear	E	P	E	E	P	6	P^{32}	6.2	0.03	4.7	3.2	1.5	
									Ba^{140}	6.7	0.1	5.7	3.8	1.9	
									I^{131}	3.7	0.02	2.0	1.8	0.2	
The Patterson Sargent Co. Cleveland, Ohio	No. 2 Special Coating 4730	Clear	E	P	E	E	P	5	P^{32}	7.0	0.01	5.0	3.9	1.1	
									Ba^{140}	6.1	0.006	3.9	3.4	0.5	
									I^{131}	2.8	0.04	1.4	1.0	0.4	
M. Shiller Co. Brooklyn, N. Y.	Enamel No. 3	White	E	P	E	E	P	5	P^{32}	4.4	0.05	3.1	3.0	0.1	
									Ba^{140}	5.6	0.06	4.4	3.9	0.5	
									I^{131}	3.5	0.01	1.5	1.4	0.1	
Phenoplast Corp. New York 17, N. Y.	Phenoplast	Clear	E	P	E	E	P	4	P^{32}	4.0	0.3	3.5	3.3	0.2	Poor resistance to impact. Peels rather easily from "Transite".
									Ba^{140}	5.1	0.06	3.9	3.2	0.7	
									I^{131}	4.1	0.005	1.8	1.7	0.1	
Sherwin-Williams Company	Lin-X Varnish	Clear	E	P	E	E	P	5	P^{32}	5.4	0.02	3.7	3.6	0.1	
									Ba^{140}	5.5	0.03	4.0	3.8	0.2	
									I^{131}	3.2	0.03	1.7	1.6	0.1	
Corrosite Corp. New York 17, N. Y.	Plastic Coating 541	Green	F	P	E	E	P	6	P^{32}	7.2	0.01	5.2	3.7	1.5	
									Ba^{140}	6.8	0.01	4.8	4.1	0.7	
									I^{131}	2.8	0.006	1.5	1.5	0.03	
Prufcoat Laboratories, Inc. New York, N. Y.	Prufcoat	Gray	E	E	G (E)	P (E)	P	5	P^{32}	5.1	0.02	3.4	3.4	0.03	Very good for floors if allowed to cure 1 week. Best results on walls and ceilings.
									Ba^{140}	5.1	0.03	3.6	3.5	0.1	
									I^{131}	4.0	0.004	1.6	1.5	0.1	
Inertol Co., Inc. Newark 5, N. J.	Rigortex 2202	Clear	F	P	E	E	P	6	P^{32}	7.0	0.01	5.0	3.5	1.5	
									Ba^{140}	6.5	0.02	4.8	3.9	0.9	
									I^{131}	3.9	0.01	1.9	1.8	0.1	
The Glidden Co. Cleveland, Ohio	No. 3 Black Coating 150-B-900	Black	P	P	E	E	P	4	P^{32}	6.0	0.04	4.6	3.4	1.2	
									Ba^{140}	3.7	0.8	3.6	3.1	0.5	
									I^{131}	2.9	0.08	1.8	1.3	0.5	

(Continued on next page)

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
The Glidden Co. Cleveland, Ohio	B6104 Alkyd	White	P	P	E	E	P	4	P^{32}	5.7	0.01	3.7	3.2	0.5	
									Ba^{140}	4.6	0.01	2.6	2.6	0.03	
									I^{131}	2.9	0.03	1.4	1.0	0.4	
Gordon-Lacey Chemical Products Company Maspeth, L.I., N.Y.	J241B	Gray	P	P	E	E	P	5	P^{32}	5.6	0.04	4.2	3.1	1.1	
									Ba^{140}	5.9	0.02	4.2	3.9	0.3	
									I^{131}	3.4	0.02	1.7	1.7	0.03	
The Glidden Co. Cleveland, Ohio	No. 2 Black Coating Y505	Black	G	P	E	G	P	3	P^{32}	3.2	0.4	2.8	2.2	0.6	
									Ba^{140}	3.4	0.2	2.7	2.2	0.5	
									I^{131}	1.0	0.4	0.6	0.5	0.1	
Lithgow Corp. Chicago 9, Ill.	LC-600 Clear	Clear	P	F	E	G	P	3	P^{32}	3.3	0.3	2.8	2.3	0.5	
									Ba^{140}	3.7	0.05	2.4	2.3	0.1	
									I^{131}	2.3	0.06	1.1	1.0	0.1	
The Patterson Sargent Co. Cleveland, Ohio	No. 1 Special Coating L-455-X	Clear	F	P	G	E	P	7	P^{32}	8.0	0.006	5.8	4.1	1.7	
									Ba^{140}	7.8	0.004	5.4	3.7	1.7	
									I^{131}	4.2	0.008	2.1	1.9	0.2	
M. B. Suydam Co.	Lead Paste and D. C. Vehicle WC-1516-E	Clear	P	P	E	E	P	3	P^{32}	4.0	0.3	3.5	3.2	0.3	
									Ba^{140}	3.2	0.06	2.0	1.8	0.2	
									I^{131}	1.5	0.4	1.1	1.1	0.01	
American Lacquer and Solvent Co. Phoenixville, Pa.	CO-Loidal-Ac L1C26-T1	Clear	P	P	E	G	P	--	P^{32}	--	--	--	--	--	
									Ba^{140}						
									I^{131}						
United Chromium, Inc.	Ucilon 451	Gray	P	P	G	E	P	4	P^{32}	3.9	0.2	3.2	2.8	0.4	
									Ba^{140}	5.2	0.005	2.9	2.6	0.3	
									I^{131}	3.6	0.008	1.5	1.0	0.5	
American Lacquer and Solvents Co. Phoenixville, Pa.	Co-Loidal-Ac L1B9-T1	Black	P	P	G	G	P	--	P^{32}	--	--	--	--	--	
									Ba^{140}						
									I^{131}						

(Continued on next page)

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
Maurice A. Knight Co. Akron 9, Ohio	Permanite FAH	Black	P	P	P	F	P	4	P^{32}	5.2	0.1	4.2	3.5	0.7	
									Ba^{140}	3.3	1.1	3.6	3.3	0.3	
									I^{131}	2.5	0.2	1.8	1.5	0.3	
Alfred Hauge and Co., Inc. Brooklyn, N.Y.	615 Rubalt	Green	P	P	P	P	P	4.0	P^{32}	4.9	0.09	3.8	2.7	1.1	
									Ba^{140}	5.0	0.04	3.6	3.3	0.3	
									I^{131}	3.3	0.02	1.6	1.4	0.2	
Corrosite Corp. New York 17, N.Y.	Plastic Coating Aluminum	Aluminum	P	P	P	P	P	4.0	P^{32}	3.6	0.5	3.3	2.7	0.6	
									Ba^{140}	4.8	0.3	4.3	3.7	1.1	
									I^{131}	3.1	0.02	1.4	1.3	0.1	
Lithgow Corp. Chicago 9, Ill.	Cotoid	Gray	P	P	P	P	P	5	P^{32}	4.4	0.2	3.7	3.1	0.6	
									Ba^{140}	5.9	0.03	4.4	3.5	0.9	
									I^{131}	3.9	0.1	1.9	1.8	0.1	
United Chromium Inc.	Ucilon 1601	Aluminum	P	P	P	P	P	5	P^{32}	5.8	0.03	4.3	3.2	1.1	
									Ba^{140}	6.1	0.03	4.6	3.9	0.7	
									I^{131}	3.4	0.02	1.7	1.6	0.1	
Koppers Co., Inc. Pittsburg 19, Pa.	Bituplastic - 28	Black	P	P	-	-	P	--	P^{32}	--	--	--	--	--	
									Ba^{140}						
									I^{131}						

TABLE 29

Strippable Coatings

Monsanto Chemical Co. St. Louis, Mo.	J653 Research Sample	Black	G	F	E	E	E	---***	P^{32}		0.07				
									Ba^{140}		0.4				
									I^{131}		0.3				

*** Spill indices were not determined for strippable coatings and tapes.

(Continued on next page)

MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TESTS			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
American Pipe and Const. Co.	Amercoat 1149	Clear	E	E	E	E	P	--	P^{32} Ba^{140} I^{131}	--	--	--	--	--	An intermediate "release" coat must be used for good stripping.
Midland Ind. Finishes Co.	Silicone L3X173	Blue	E	E	E	E	P	--	P^{32} Ba^{140} I^{131}		0.03 0.04 0.03				
U.S. Stoneware	Tygofilm TP217	Clear	E	E	E	E	P	--	P^{32} Ba^{140} I^{131}		0.004 0.008 0.02				
Atlas Powder Co. North Chicago, Ill.	Brevon 536-2-253	Black	E	E	E	E	P	--	P^{32} Ba^{140} I^{131}		0.001 0.007 0.004				
Maas and Waldstein Company	Coopeel 97	Clear	E	E	E	E	P	--	P^{32} Ba^{140} I^{131}		0.001 0.03 0.2				
R.M. Hollingshead Corp. Camden, N.J.	G. E. Cocoon	Clear	E	E	E	E	P	--	P^{32} Ba^{140} I^{131}		0.4 0.3 4.7				Very good used extensively at ORNL
Gordon Lacey Chemical Products Co. Maspeth L. I., N. Y. C.	J220F	White	E	E	E	E	P	5	P^{32} Ba^{140} I^{131}	5.9 5.4 2.8	0.02 0.03 0.005	4.2 3.9 0.5	3.0 2.3 0.4	1.2 1.6 0.1	
Gordon Lacey Chem. Prod. Co. Maspeth, L. I., N. Y. C.	A89A	Black	E	E	E	E	P	5	P^{32} Ba^{140} I^{131}	6.3 5.8 3.3	0.04 0.01 0.02	4.9 3.8 1.6	3.1 3.2 1.5	1.8 0.6 0.1	
American Resinous Chem. Co. Peabody Mass.	Liquid Strip 553-45A	Clear	G	G	E	E	P	--	P^{32} Ba^{140} I^{131}		0.1 0.3 0.03				

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MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TEST			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
Bisonite Co., Inc.	751	White	P	G	G	E	P	--	P^{32} Ba^{140} I^{131}		0.009 0.03 0.01				
Spraylat Corp. New York, 16, N.Y.	Spraylat SC-1054	Clear	G	P	F	P	G	--	P^{32} Ba^{140} I^{131}		0.1 0.3 0.3				
B.F. Goodrich Chem. Co. Cleveland 15, Ohio	Geon Latex 31-X	Clear	P	P	P	E	P	--	P^{32} Ba^{140} I^{131}		21.0 21.0 6.0				

TABLE 30

Baked Coatings

United Chromium Inc. Detroit 7, Michigan	Unichrome B-124-1	Gray	E	P	G	G	E	3	P^{32} Ba^{140} I^{131}	3.5 3.4 1.8	0.04 0.03 0.08	2.1 1.9 0.7	1.9 1.8 0.6	0.2 0.1 0.1	
Interchemical Corp. Cincinnati Ohio	583 Polytherm (Silicone)	White	G	P	E	G	P	8	P^{32} Ba^{140} I^{131}	9.2 8.9 5.2	0.002 0.002 0.003	6.5 6.2 2.7	4.5 5.1 2.6	2.0 1.1 0.1	
du-Pont Wilmington 98, Delaware	Shell Varnish Enamel	Clear	G	P	G	F	E	7	P^{32} Ba^{140} I^{131}	7.0 9.1 4.0	0.008 0.004 0.01	4.9 6.7 2.0	4.0 4.0 1.9	0.9 2.7 0.1	
Interchemical Corp.	575 Polytherm (Silicone)		G	P	G	G	P	7	P^{32} Ba^{140} I^{131}	8.0 7.3 5.1	0.006 0.003 0.006	5.8 4.8 2.9	4.9 4.7 2.8	0.9 0.1 0.1	

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MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TEST			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
United Chromium, Inc.	Unichrome B-124	Clear	F	P	G	F	E	6	P^{32}	6.3	0.05	5.0	3.5	1.5	
									Ba^{140}	7.3	0.02	5.6	3.8	1.8	
									I^{131}	4.3	0.004	1.9	1.9	0.04	
Atlas Powder Co.	Duranite H	White	P	P	E	G	P	6	P^{32}	6.8	0.005	4.5	3.4	1.1	
									Ba^{140}	5.7	0.02	4.0	2.4	1.6	
									I^{131}	4.1	0.07	1.9	1.7	0.2	
Interchemical Corporation	60-9 Polytherm (Silicone)	Clear	P	P	P	P	P	6	P^{32}	7.0	0.01	5.0	4.2	0.8	
									Ba^{140}	6.2	0.005	3.9	3.4	0.5	
									I^{131}	4.9	0.007	2.7	2.6	0.1	
Stanley Chem. Co. East Berlin, Conn.	Black Plasticol 77R-88	Black	Could not be applied					--	P^{32}	3.4	1.1	3.4	2.7	0.7	
									Ba^{140}	2.5	1.4	2.5	1.9	0.6	
									I^{131}	2.1	0.2	1.4	0.3	1.1	

TABLE 31

Miscellaneous Materials

Atomic Energy Commission Oak Ride, Tenn.	Fluorthene (Flame Sprayed)	Clear	E	E	E	E	E	4	P^{32}	5.7	0.1	4.7	3.8	0.9	Spill index-flame sprayed plaques. Chem. Resist. - not flame sprayed.
									Ba^{140}	4.7	0.5	4.4	2.9	1.5	
									I^{131}	2.0	1.0	2.0	1.5	0.5	
Dow Chem. Co. Midland, Mich.	Polythene	Clear	E	E	E	E	E	6	P^{32}	7.4	0.009	5.3	3.8	1.5	
									Ba^{140}	6.1	0.9	6.0	4.0	2.0	
									I^{131}	4.7	0.03	3.2	2.8	0.4	
General Electric Co.	Textolite	Black	E	E	E	E	E	4	P^{32}	5.0	0.07	3.8	3.2	0.6	
									Ba^{140}	4.9	0.05	3.6	3.4	0.2	
									I^{131}	3.0	0.03	1.5	1.2	0.3	

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MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TEST			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D.I.	Step 1 D.I.	Step 2 D.I.	
E. H. Sheldon Co., Muskegon, Michigan	Shelstone	Black	E	E	E	E	E	3	P^{32}	5.3	0.004	2.9	2.5	0.4	
									Ba^{140}	1.2	21.6	2.5	2.3	0.2	
									I^{131}	1.6	0.07	0.4	0.3	0.1	
Sloane and Blabon Co., New York, N.Y.	Sheet Flooring 386-D	Clear	E	E	E	E	E	5	P^{32}	5.6	0.08	4.5	3.6	0.9	
									Ba^{140}	5.7	0.07	4.5	3.4	0.1	
									I^{131}	3.4	0.005	1.1	1.0	0.1	
Sloane and Blabon Co., New York, N.Y.	Sheet Flooring 386-E	Gray	E	G	E	E	E	4	P^{32}	2.9	0.6	2.7	2.6	0.1	
									Ba^{140}	5.0	0.1	4.0	2.3	1.7	
									I^{131}	3.4	0.007	1.2	1.0	0.2	
Sloane and Blabon Co., New York, N.Y.	Sheet Flooring 386-A	Cream	E	E	E	E	P	3	P^{32}	3.8	0.5	3.5	3.4	0.1	
									Ba^{140}	3.3	0.08	2.2	2.0	0.2	
									I^{131}	1.6	0.08	0.5	0.5	0.02	
Sloane and Blabon Co., New York, N.Y.	Sheet Flooring 386-C	Cream	E	E	E	E	P	3	P^{32}	3.7	0.5	3.4	3.2	0.2	
									Ba^{140}	3.1	0.4	2.7	1.9	0.8	
									I^{131}	1.9	0.07	0.7	0.7	0.02	
B.F. Goodrich Company	Koroseal Flooring	Blue	E	E	E	E	P	3	P^{32}	2.2	2.4	2.5	2.3	0.2	
									Ba^{140}	3.0	0.4	2.6	2.1	0.5	
									I^{131}	3.0	0.04	1.6	1.3	0.3	
Rom and Haas Knoxville, Tenn.	Lucite	Clear	E	E	E	E	P	7	P^{32}	9.4	0.005	7.1	3.6	3.5	
									Ba^{140}	5.2	0.3	4.7	4.4	0.3	
									I^{131}	5.6	0.002	2.9	2.6	0.3	
Bauer and Black Chicago 16, Ill.	Polyken Tape	White	E	G	E	E	P	--	P^{32}		0.08				
									Ba^{140}		0.05				
									I^{131}		0.07				
Goodyear Rubber Company, Akron, Ohio	Vinyl Style 3511 Flooring	Gray	E	G	E	E	P	2	P^{32}	2.9	5.3	3.4	3.3	0.1	
									Ba^{140}	1.6	5.6	2.4	2.0	0.4	
									I^{131}	2.2	0.2	1.5	1.4	0.1	

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MANUFACTURER	TRADE NAME	COLOR	CHEMICAL RESISTANCE TESTS					CONTAMINATION TESTS							REMARKS
								CONTAMINATION RESISTANCE			SUSCEPTIBILITY TEST	DECONTAMINATION TEST			
			3 M HNO_3	3 M NaOH	3 M H_2SO_4	3 M HCL	Hexone	*Gross Av. Spill Index	Isotope	Spill Index	% Adsorbed in 1 Hr.	Overall D. I.	Step 1 D.I.	Step 2 D.I.	
Sloane and Blabon Co. New York, N.Y.	Sheet Flooring -386-B	Brown	E	P	E	E	P	3	P^{32}	3.6	0.3	3.1	2.9	0.2	
									Ba^{140}	4.4	0.1	3.4	2.0	1.4	
									I^{131}	1.9	0.1	0.9	0.7	0.2	
Industrial Tape Corp. New Brunswick, N.J.	Jonflex Tape	White	E	P	E	E	P	--	P^{32}		0.5				
									Ba^{140}		18.0				
									I^{131}		0.2				
Industrial Tape Corp. New Brunswick, N.J.	Olive Drab -68 Tape	Green	P	P	E	G	P	--	P^{32}		9.9				
									Ba^{140}		43.1				
									I^{131}		41.1				
Industrial Tape Corp. New Brunswick, N.J.	Acetate Fiber Permacel 99 (Tape)	Clear	P	P	G	P	P	--	P^{32}		0.1				
									Ba^{140}		0.1				
									I^{131}		0.01				
Mastic Tile Corp. of America	Asphalt Flooring	Brown	P	P	P	P	P	2	P^{32}	1.7	5.2	2.4	2.1	0.3	
									Ba^{140}	1.9	14.2	2.9	2.1	0.8	
									I^{131}	1.5	16.5	2.8	2.8	0.02	

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TABLE 32

Structural Materials

MATERIAL	GROSS AVERAGE SPILL INDEX	ISOTOPE	SPILL INDEX	PERCENT ADSORBED IN ONE HOUR	OVERALL D. I.
Polished Stainless Steel Type No. 347	7	P^{32}	3.7	0.3	3.2
		Ba^{140}	7.3	0.04	5.7
		I^{131}	8.7	0.007	6.5
Pyrex Glass	6	P^{32}	5.9	0.02	4.2
		Ba^{140}	4.8	0.2	4.1
		I^{131}	6.4	0.04	5.0
Bonded Stainless Steel Type No. 302	5	P^{32}	3.9	0.6	3.7
		Ba^{140}	5.9	0.4	5.5
		I^{131}	4.9	0.05	3.6
Micro Rold Stainless Steel - Type No. 302	5	P^{32}	4.2	0.5	3.9
		Ba^{140}	6.2	0.2	5.5
		I^{131}	4.9	0.1	3.9
Unpolished Stainless Steel - Type No. 316	4	P^{32}	2.5	0.8	2.5
		Ba^{140}	4.0	0.8	3.9
		I^{131}	6.1	0.06	4.9
Lead	3	P^{32}	2.9	80	4.8
		Ba^{140}	3.9	2	4.2
		I^{131}	3.6	3	4.1
Aluminum		P^{32}		0.5	
		Ba^{140}		0.04	
		I^{131}		0.05	
Structural Steel		P^{32}		76.6	
		Ba^{140}		9.3	
		I^{131}		0.1	

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MATERIAL	GROSS AVERAGE SPILL INDEX	ISOTOPE	SPILL INDEX	PERCENT ADSORBED IN ONE HOUR	OVERALL D.I.
Transite		P^{32}		95.0	
		Ba^{140}		98.0	
		I^{131}		9.7	
Plywood		P^{32}		48.0	
		Ba^{140}		82.0	
		I^{131}		41.0	
Concrete (Special Floor Sample)		P^{32}		95.4	
		Ba^{140}		90.3	
		I^{131}		20.2	

(1) Tompkins, P.C., Bizzell, O.M., "Radioactive Decontamination of Laboratory Surfaces", ORNL 381 - In Press

(2) Tompkins, P.C., Bizzell, O.M., Watson, C.D., "Radioactive Decontamination of Laboratory Surfaces", ORNL 382 - In Press

NEW FACILITIES—ARCHITECTURAL DEVELOPMENT AND PROGRESS

The Semi-Works layout, as developed in May, has been revised but little since that time. The Austin Company has just completed its preliminary drawings, showing these facilities attached structurally to the Radio-Isotope Research facilities. The net area of the present Semi-Works is approximately 4100 ft^2; the proposed new Semi-Works net area is about 19.500 ft^2. The new facilities will provide sufficient space and equipment to handle with facility such problems as can now be visualized. The Semi-Works will have two cells 8 ft × 8 ft in area, 27 ft high, with 10 ft deep pits, 4 ft × 8 ft in area, in the bottom of these two. Two other cells, 8 ft × 14 ft in area will also be 27 ft high; these can both be divided into two cells each, 8 ft × 6 ft in area. All of the cells in this bank can be extended upward for another 10 ft, by using concrete block. It will be seen that complete flexibility of use will be herein obtained, both horizontally and vertically.

The earlier scheme for permanentization of the 205 building was finally discarded with the acceptance of a layout which is more desirable.

There will be facilities of the following nature provided:

(1) 2 Radiochemical labs of 575 ft^2 each,
(2) 2 Control labs of 575 ft^2 each,
(3) 1 Dilution and Make-up lab of 325 ft^2,
(4) 1 Pilot Plant Warm lab of 325 ft^2,
(5) 1 Solution and Equipment area of 3500 ft^2,
(6) Office space of 1650 ft^2,
(7) Operating gallery behind entire cell block.

The existing counting room and cells will remain. Clothing change and wash facilities will be shared with Bldg. 105 in a new structure.

Plans for office and shop space for the 706-HB building have not progressed since the last quarterly report. This, however, is still under consideration, and will be developed at the earliest opportunity.

Design criteria for the New Research Building have been completely developed and transmitted to the Austin Company. Studies are still being made of several alternates for cost purposes. The desired layout will provide the Technical Division Section I with 1 glass and bottle washing room, 6 "cool" labs, 4 "hot" labs, 2 alpha labs, and 4 labs for Technical Division analytical

work. There will also be 1 laboratory for use of Section II personnel. The foregoing 18 rooms will all be about 24 ft × 24 ft in size. In addition, there will be 2 counting rooms, and other service facilities, including office space sufficient to provide for the 18 labs above listed.

SOURCE AND FISSIONABLE MATERIAL SURVEY

At the request of the Operations Division, a survey of all source and fissionable materials is being made. The purpose of the survey is to list all phases of ORNL operations and research in which source and/or fissionable materials are handled, to determine the quantities of such materials involved, and to outline and evaluate the effectiveness of an economically sound, integrated material control system.

The materials handled in ORNL that are subject to material balance control are (1) natural or slightly depleted uranium, (2) enriched U^{235}, (3) plutonium, (4) U^{233} and (5) thorium.

In general an effective material control system will recognize the following internal balances: (1) overall plant balance, (2) balance across slugs irradiated in ORNL or other reactors, (3) material balances across unit processes or experiments, and (4) waste system balance.

The inherent accuracies in an analytical material balance system must be taken into account, and the obtainable accuracies must be weighed against the cost in money and operational difficulty to accomplish them. Limiting accuracies in any material control system using radioactive material are imposed by the following factors: (1) accuracy of slug calculations—initial loading, irradiation, (2) sampling accuracies—nature of solution, type of samples, blending of solution, timing, (3) accuracy of measurement of liquid level and specific gravity in vessels or lines from which samples are taken, and (4) analytical accuracy for the material in the mixture in which it is contained.

The survey will attempt to evaluate all of these factors plus physical conditions such as the hazard of obtaining the sample and its analysis, number of personnel and amount of laboratory space to carry on the program, and estimated cost of the program. It is hoped that a satisfactory material balance system that is a hindrance from neither an economic nor an operational standpoint can be evolved.

PERSONNEL

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F. L. Steahly*, *Chief, Section I - Chemical Process Development*

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T. C. Runion
W. E. Tomlin
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L. E. Morse
L. A. Byrd
W. B. Howerton
E. R. Johns

R. E. Leuze, *Group Leader - Dry Fluoride*

TECHNICIANS

C. P. Johnston
H. B. Graham

Special Assignment Group

TECHNICIANS

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Arlene Kibbey
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J. M. Delozier
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I. R. Higgins, *Group Leader - RaLa*

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W. H. Lewis - *Group Leader - "25"*

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J. B. Ruch - *Metal Recovery*

C. D. Watson, *Group Leader - Testing and Design*

TECHNICIANS

G. A. West
G. Jones

W. R. Winsbro, *University of Virginia, Summer Employee*

E. J. Freeh, *Summer Employee*
W. P. Jensen, *Summer Employee*
R. N. Saleeby, *Summer Employee*

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D. G. Reid, *Chief, Section VI - Pilot Plants*

H. K. Jackson, *Supervisor - Redox Process Data*

E. L. Nicholson - *Waste Disposal*

A. M. Rom, *Supervisor - 25 Process Data*

N. J. Rigstad, *Chief Supervisor - Pilot Plants*

J. W. Landry - *Trainee*

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