



3 4456 0360244 8

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION



ORNL-45
Progress Report

Op. 7a

OAK RIDGE NATIONAL LABORATORY

OPERATIONS DIVISION REPORT

for

Month Ending April 30, 1948

FILE OPERATIONS
SEPARATIONS
RADIOISOTOPES

by

M. E. Ramsey, E. J. Witkowski, A. F. Rupp, and L. B. Emlet

Date Received: 5/13/48

Date Issued: 5/13/48



OAK RIDGE NATIONAL LABORATORY
CENTRAL RESEARCH LIBRARY
CIRCULATION SECTION
2050N ROOM 175
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this
report, send in name with report and
the library will arrange a loan.
NCR-2050 3-77

CLASSIFICATION CHANGED TO:

DECLASSIFIED

BY AUTHORITY OF: T-10-1149

BY: P. Morrison 7/8/57

DISTRIBUTION

1. G. T. Felback
- 2-4. 706-A
5. 706-B
6. Biology Library
7. Training School
- 8-12. Central Files
13. E. L. Murphy
14. C. Rucker
15. Central Files
16. M. D. Peterson
17. A. M. Weinberg
18. A. Holdender
19. E. H. Taylor
20. H. Etherington
21. E. A. Bailey
22. J. A. Cox
23. A. F. Rupp
24. D. G. Reid
25. M. E. Ramsey
26. E. J. Witkowski
27. L. B. Emlet
28. J. A. Swartout
29. Library Branch, Technical Information Division
- 30-32. AEC, Washington



SUMMARY

1. Pile operations were normal for the month.
2. Excess pile reactivity is about sixty to seventy inhours. The increase of twenty inhours resulted when the water was drained from the Hole 19 tube.
3. A total of 6,900 $\text{Ca}(\text{NO}_3)_2$ cans (about 1,800 pounds) are now in the pile for Cl^{14} production.
4. The bearings on the #2 fan were replaced. During this time the pile was operated at half power.
5. RaLa run #25, originally scheduled to start May 15, 1948, has been postponed for one month at the request of Los Alamos.
6. The RaLa process equipment has been reconditioned and will be tested during the next month.
7. Isotope production continued satisfactorily.
8. The all-column fission product separation equipment is being modified and all stainless steel replaced with glassware.
9. The 269 shipments made this month is a decrease of ten percent over last month. A total of 3,209 shipments have been made since the start of the program in August, 1946.

A. 100 AREA OPERATION

I. Operating Data:

	<u>April</u> <u>1948</u>	<u>March</u> <u>1948</u>	<u>Year to Date</u>
Total Accumulated KWH-----	2,541,495	2,707,200	10,051,594
Average KWH/operating hour-----	3693.34	3906.78	3787.72
Average KWH/24-hour day-----	3529.85	3638.71	3462.48
Percent Lost Time-----	4.3%	6.9%	8.6%
Approx. Excess Pile Reactivity---	60-70 inhours	40-50 inhours	
Slugs Charged-----	11	463	2504
Slugs Discharged-----	11	490	2597
Product Made (grams)-----	92.76	98.80	366.85
Product Discharged (grams)-----	0.03	4.62	53.73

II. Pile Operation:

Pile operation was normal throughout the month with the usual shutdowns for removal and insertion of samples and other miscellaneous work.

The excess pile reactivity is sixty to seventy inhours or about twenty inhours above the excess reactivity for March, 1948.

During the month one of the safety rods was found to be hanging eighteen inches too low when in the withdrawn position and the two control rods were found to be out of agreement with the Selsyn recorders by about two feet. The few inhours realized, as a result of these corrections, were consumed by loading twenty-five channels with $\text{Ca}(\text{NO}_3)_2$ slugs for Cl^{14} production. The pile is now charged with the full quota of 6,500 cans of calcium nitrate to be used in Cl^{14} production.

In order to get better slow neutron flux, the slugs in the nineteen metal channels directly beneath the animal tunnels were repositioned so as to have the metal as far away from the tunnel as possible without discharging any slugs. The cadmium ratio is now approximately 200

SECRET

compared to a ratio of approximately sixty before the slugs were repositioned.

The wooden rods on the ends of the front face plugs which face metal were visually inspected during the month and found to be in good condition.

An additional stringer section was placed in Hole 14 replacing a blank graphite section giving ten additional positions for sample exposure ranging from twenty percent to twenty-seven percent of maximum neutron flux. This section was added to widen the flux range in Stringer 14.

Eleven X-slugs were installed in metal channel 1964 and their temperatures elevated by restricting the air flow through the channel. Temperatures are being taken by thermocouples on the center and the two end slugs. The west slug has been maintained at a maximum of 3500 C. with the middle slug being 2950C. and the east slug being 2300C. This work is being done by the Physics Division in order to evaluate the slug jacket life at elevated temperatures. At the present, the maximum operating temperature of slugs in the pile is 2500C. The results of this test will determine if it is feasible to increase the present operating temperatures.

III. Fan Operation:

The bearings on No. 2 fan were changed on April 21, 1948. The fan was shut down for forty-four hours. It required thirty-two hours of decontamination before the radiation level was reduced sufficiently for maintenance work to be started. The abnormally high level of radiation from the fan was due chiefly to the rupture and discharge

SECRET

~~SECRET~~

of the thirteen slugs from metal channel 2079 during the early part of December, 1947.

The bearings that were removed from the fan had badly scarred races and several marked balls.

The rotors of the oil pumps on the No. 3 fan lubrication system were found to be badly worn during the regular inspection period and were replaced.

IV. Radioisotopes:

The following table is a record of the radioisotope and research samples inserted in the pile during March and April, 1948:

	<u>MARCH</u>		<u>APRIL</u>	
	<u>Research</u>	<u>Radioisotopes</u>	<u>Research</u>	<u>Radioisotopes</u>
Stringers 13, 14, and 16	49	108	29	102
Hole 22 (Pneumatic Tube)	59	0	35	0
All other holes	<u>17</u>	<u>8</u>	<u>13</u>	<u>10</u>
TOTAL BY GROUPS	<u>125</u>	<u>116</u>	<u>77</u>	<u>112</u>
TOTAL FOR MONTH	241		189	

At the end of April, 1948, there were 392 cans of target material in Stringers 13, 14, and 16, compared to 346 cans of material in these stringers at the end of March, 1948.

V. Miscellaneous:

The calcium nitrate canning program is scheduled for completion during May, 1948. All of the calcium nitrate has been ground and pressed into pellets. These are approximately 1,600 pellets (two pellets per can) to be canned with 325 finished slugs in stock.

~~SECRET~~

VI. Experimental Work:

The Technical Division is making arrangements for borrowing equipment to take several core samples from the concrete shielding of the pile. These samples will be used to determine the structural strength of the concrete after several years' exposure to neutrons.

B. CHEMICAL SEPARATIONS DEPARTMENT

I. Radioisotopes:

1. Iodine (I¹³¹ - 8d)

Thirty-two, seventy-five-gram, cans of irradiated tellurium were processed and approximately 3,583 millicuries of I¹³¹ were shipped. All of the product was within specifications.

Iodine Development Work

A run with dead slugs was made in the tantalum-lined dissolver to continue tests on the efficiency of the removal of aluminum coatings with ten-percent HNO₃, containing 0.05% Hg(NO₃)₂ catalyst. There is some evidence that a slight mercury contamination of the UNH solution occurs. If necessary, a portion of the uranium will be dissolved in seventy-percent HNO₃ and discarded to prevent any possible Hg or Al contamination.

A request was made by the Chemistry Division for some service dissolvings of hot Hanford slugs. A series of tests were performed

~~SECRET~~

on dead slugs in glassware which showed that the bonding on Hanford slugs can be removed only with caustic solution, thus making it impossible to do this work in the tantalum-lined dissolver.

The 706-C underground waste tank was emptied to the Tank Farm and rinsed. The new venting system recently installed on the tank worked well and the operation went very smoothly.

A training program for 706-D personnel will be started with the next hot run in the dissolving and primary iodine recovery equipment.

Project W-50 P-176 (P), covering the installation of equipment in Room 10 for iodine purification, was closed during the month. All major equipment has now been fabricated. It is hoped that process testing can be started in the next month.

2. Phosphorus (P^{32} - 14.3d)

Eight, 2,000-gram, cans of irradiated sulfur were processed. Approximately 3,232 millicuries of P^{32} were shipped.

The equipment installed last month for loading sulfur cans into the melter has cut the direct radiation to tolerance levels. Present indications are that this equipment has also stopped the air contamination during loading of the sulfur cans into the melter; however, further tests are being made.

3. Carbon (C^{14} - 5100y)

No runs were made.

Cl¹⁴ Development Work - (From Be₃N₂)

The installation of facilities for Cl¹⁴ - Be₃N₂ work in Building 204 Annex is practically complete. Process equipment, such as special glassware, furnaces, and instruments which have been ordered, have not been received; this material is scheduled for installation in May, 1948.

Experimental work has gone forward in testing a number of Ba(OH)₂ absorber-precipitators. A system is being devised whereby dry BaCl₄O₃, which is quite dangerous to handle, need not be scraped off filter discs. The BaCO₃ is being handled as a slurry, centrifuged, and dried in the final container.

4. Sulfur (S³⁵ - 87.1d)

No S³⁵ was separated during the month, there being an adequate supply on hand.

5. Fission Products

UNH solution carried over from the last dissolver run was passed through the all-column equipment, mainly to secure the Zr-Cb and Y fractions.

It has been decided to decontaminate the cell and completely revise and simplify the equipment. The original equipment had been installed using stainless steel in many places, including the head tank. To prevent corrosion by the sulfuric acid used to remove uranium, one-percent HNO₃ had to be added. It was found that even this small amount of HNO₃ injures Amberlite resins over a period of time. Furthermore, HCl cannot be used, which seriously limits the flexibility of the equipment. The equipment

will be re-installed, using only glass and plastic. In the revised flowsheet, Zr-Cb will be absorbed on a small non-ion exchange pre-column and most of the remaining fission products and uranium will be passed to the large four-inch column. After the uranium is removed with sulfuric acid, all the remaining activity is removed from the column with 6-10 N HCl. Most of the HCl is distilled off this eluant, the residue is diluted with water, and it is passed through a long, thin, column; the activity concentrating in a tight band at the top of the column. The activities are then fractionated from this column with citrate solutions at various pH's. In the original system, citrate solution was used to elute material from the four-inch column, HNO₃ was added to destroy the complexing action of the citrate, and the activities were placed on several small columns. However, tight bands of activity are not formed at the tops of the columns by this method and the subsequent fractionation is very poor.

About 150 millicuries of yttrium was purified during the month and is ready for shipment. The limit for alpha contamination was changed from 70 c/min/mc Y to 150 c/min/mc Y because the former value is too low to be consistently counted in the presence of the large amount of beta activity.

The purification process for Zr-Cb has been worked out and extraction equipment for one-half-curie batches is being set up. The Zr-Cb is extracted in 0.5 M TTA-benzene, the Pu washed out with dilute HCl, the Cb extracted with strong HCl, and most of the TTA is removed from the organic phase with sodium hydroxide. The

~~CONFIDENTIAL~~

amount of complexing agent in the organic phase is then so small that the tightly-held Zr may be extracted with HCl-oxalic acid solution.

Two curies of Ce^{144} are in the process of purification, being separated mainly from Pu. This material will be ready during the first of May, 1948.

6. Ruthenium (Ru^{106} - 1y)

The extensive changes in the precipitation equipment located in the Tank Farm Area have been completed. The additional shielding provided has been found adequate for safe operation.

Forty concentrations, using 27,600 gallons of metal waste supernatant, have been made since the equipment changes were completed.

About 150 millicuries of Ru^{106} were purified during the month. Some trouble with yields was experienced by the regular operating group in the primary separation of Ru from the Tank Farm wastes on ferrous hydroxide. A study showed that it is necessary to use the pure grade of ferrous sulfate originally used in working out the process and that the sample of supernatant used to check yield must be drawn from a point high up in the tank, well away from the settled ferrous hydroxide.

The Ru distillation equipment is being revised and more shielding added to permit safer operation.

A method is being sought to re-carry the Ru^{106} from the accumulated ferrous hydroxide precipitates, thus avoiding distillation from large volumes of liquid. Precipitation of copper sulfide

in a sulfuric acid solution of ferrous salt and Ru seems promising, an eighty percent yield being obtained in the first experiments.

7. Calcium (Ca⁴⁵ - 180d)

An adequate supply of high-specific activity Ca⁴⁵ is on hand. Remote control equipment is being fabricated for the scandium carrier-free Ca⁴⁵ method.

8. Strontium (Sr⁸⁹⁻⁹⁰ - 30y, 55d)

Three runs were made this month. The first two yielded approximately 3,700 millicuries with approximately sixty-five percent of it being Sr⁹⁰. Analytical results have not been received on the third run.

9. Iron (Fe⁵⁵⁻⁵⁹ - 4y, 44d)

No further work this month. Samples will arrive from Hanford during May, 1948.

10. Miscellaneous Materials

A unit of tantalum was dissolved by fusion in K₂CO₃. There is a solution of about sixty millicuries on hand.

A sample of tin was dissolved containing approximately three millicuries of Sb¹²⁵. This solution also contains a small amount of tin activity.

A unit of mercury was dissolved but has not yet been analyzed.

A Hanford-irradiated sample of KCl was opened and samples prepared for customers.

~~SECRET~~

B. CHEMICAL SEPARATIONS DEPARTMENT

II. Barium (Ba140 - 12.5d)

Los Alamos reported having difficulty in making the first separation of our product (Run #24) which was shipped on March 13, 1948. The three subsequent separations were satisfactory.

It is suspected that the cause of their troubles may have been the speck of foreign material which was observed through a periscope, to be lying on the shoulder of the cone prior to the removal of the product from our cells. This material was blown into the carrier by the cell ventilation air. It was impossible to remove it at that stage of the operation.

Repairs and alterations of Cell B equipment continued during the entire month. The following work has been completed:

1. The cubicle glassware and Tygon lines were completely repaired and tested.
2. The electrolytic cell was repaired, rewired, replaced, and tested under simulated operating conditions.
3. New stainless steel tank supports and braces were installed under three of the Cell B tanks (B-1; B-5; and B-10).
4. The overflow line from the evaporator (B-6) was blanked off to improve the off-gas ventilation.
5. New wheel bearings were placed on the product carrier dolly and the dolly was realigned to give free movement on the tracks.

6. All Tygon lines in Cell B were replaced with 25-12 stainless steel pipe with extra heavy Tygon connections between the pipe ends and tanks.
7. All lines were tested for leaks and repaired wherever necessary.
8. The plug hook and chimney guide rods were straightened.
9. The cell was repainted and relamped.
10. The final evaporator cone elevator was completely redesigned and reinstalled to give free movement of product assembly
11. A special wall plug with an adjustable bulb was installed to provide more light when setting the product carrier on the dolly.

All major repairs have been completed. A final check of all cell equipment will be made before the cell door is closed.

Run #25, originally scheduled to start about the 15th of May, was again postponed at the request of Los Alamos. It is now tentatively scheduled to start about the middle of June, 1948.

The study to use Hanford-irradiated slugs was continued. The preliminary design of the slug carrier was approved by the General Electric Company. The detailed design work has been completed and the fabrication of one carrier started. The completion of this carrier is expected in one month.

III. Tank Farm and Burial Ground:

1. Tank Farm

- (a) The W-3 tank was put into service for storage of the aluminum-plutonium waste from the Hot Pilot Plant. The two inches of uranium waste slurry remaining in the tank could not be pumped out completely.
- (b) High waste activity at the Retention Pond was found during the month. The drywell at W-11 tank was first suspected, but after further investigation, a bad flange gasket in a valve pit was discovered. The leak occurred in the transfer line from W-12 tank to W-5 chemical waste storage tank. After the installation of new gaskets, the Retention Pond activity dropped back to normal.
- (c) While looking for the source of activity in the Retention Pond, a leak was found in the transfer line from A-6 tank in 706-D to W-9 metal waste storage tank. This leak occurred in the jet pit in the Tank Farm. At the end of the month decontamination of the pit and lines involved was underway. Repairs will be made as soon as the activity is reduced to a safe level.
- (d) Repair orders have been issued to rebuild the float gauges on all the tanks. The present gauges are in poor condition. Floats have been removed from tanks W-5, W-7, W-9, W-10, and W-11, and should be rebuilt and back in service soon. The remainder will then be reworked.

- (e) The J. A. Jones Construction Company has laid a two-inch stainless steel line from the new solvent recovery building, south of 706-A, to the W-5 chemical waste tank. It was tied into W-5 by the Maintenance Department. The line has been air tested and covered with earth.
- (f) The project to install the new stainless steel W-12 waste tank is complete and in operation. W-11 tank is now used only as an overflow tank from W-12.
- (g) A concrete ramp was built under the "A" frame at W-4 tank to accommodate the large trucks coming into Oak Ridge National Laboratory from Chicago. This pad facilitates the unloading of pots and drums and also aids in the decontamination of any possible spills of radioactive material.
- (h) The pole replacement program in the Tank Farm is about ninety percent complete.
- (i) In the past it has been necessary to remove the earth from above the concrete inlet diversion box to W-1 and W-2 tanks approximately every two or three months to remove accumulated trash from the strainers. Several times the box has overflowed and contaminated the surrounding ground. To prevent this overflow and to facilitate the cleaning of the strainers, the concrete walls of the box were extended to the top of the ground and the metal top plate bolted to the new concrete. This will shorten cleaning time considerably.

~~SECRET~~

- (j) Approximately 27,600 gallons of supernatant from the precipitated metal in W-7 tank was used in the ruthenium separation process this month. This liquid contained an average of .0057% uranium.
- (k) The Hot Pilot Plant transferred 60.98 kg of uranium to W-4 metal storage tank.
- (l) Twenty-one pots and fifteen drums of waste were received from Chicago this month. One pot contained 7.14 kg of uranium; the remainder had only aqueous solutions. Two stainless steel drums contained 52.14 kg of uranium; six were aqueous solutions and five contained hexone. Two drums were black iron and were buried at the Burial Ground.
- (m) The 706-C Building transferred 1,500 gallons of metal waste to W-9 tank.
- (n) The following is a listing of the movement of liquids in the Tank Farm for April, 1948:

WATER WASTES

<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Apr.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-1 & 2	8,800 gal.	380,000 gal.	380,000 gal.	White Oak Creek & W-5	8,800 gal.

Approximately five percent of the 380,000 gallons was routed to W-5 tank. The remainder went to the Settling Basin.

~~SECRET~~

CHEMICAL WASTES

<u>Tanks</u>	<u>Gallons Capacity</u>	<u>Est. Amt. Rec'd-Apr.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-5 & 6	340,000	186,000 gal.	109,800 gal.	Settling Basin	28,800 gal.

METAL WASTES

W-3-4-7- 8-9-10	755,700	3,000	27,600	Ru Separation Process	165,334 gal.
--------------------	---------	-------	--------	-----------------------	--------------

SETTLING BASIN

<u>Total Est. Discharge</u>	<u>Total Curies Discharged</u>	<u>Beta Counts/min/ml</u>			<u>Discharged To</u>
		<u>Average</u>	<u>High</u>	<u>Low</u>	
20,862,000 gal.	77.22	325	1372	61	White Oak Creek

EAST AND WEST POND

2,782,000 gal.	5.22	150	250	57	White Oak Creek
----------------	------	-----	-----	----	-----------------

RETENTION POND

440,800 gal.	7.72	146	95,115	12	White Oak Creek
--------------	------	-----	--------	----	-----------------

2. Burial Ground

a. Special Burials

1. Four alpha contaminated, Dayton shipments.
2. Three drums of liquid waste from Semi-works.
3. Four drums of liquid from Cell 4, Building 205, emptied into alpha trench.
4. One hot plate from Room 25, Building 706-A.

b. Stored at Burial Ground

Small stainless steel tank from Semi-works.

c. Removed from Burial Ground

1. Four, fifty-five-gallon, stainless steel drums to the Semi-works.
2. Two Bird, twelve-inch, bowl centrifuges to the K-25 Area.

d. Routine Burials

Three hundred and thirteen red cans of contaminated trash from the Restricted Area.

C. RADIOISOTOPE PRODUCTION AND SHIPMENTS

General:

The following table indicates the number of shipments for March and April, 1948, and a total-to-date figure since August, 1946, the start of the Isotope Distribution Program:

	MARCH 1948	APRIL 1948	TOTAL AUGUST, 1946, to APRIL, 1948, Inc.
Separated Material 706-D Area	221	211	2,301
Unseparated Material 100 Area	78	58	908
TOTAL	299	269	3,209

Two hundred and eleven of the monthly shipments were to off-project customers, thirty-one to other Atomic Energy Commission installations, and twenty-seven to foreign countries.

Most isotope shipments are made on Tuesdays and Wednesdays; since the month of April had two less shipping days than March, the quantity of material distributed was also less.

In order to make the shipment of radioisotopes to foreign countries less expensive, it has been decided to raise the radiation limit on rail and boat shipments from 15 mr/hr to a maximum of 200 mr/hr. For the present this will be done only in special cases where an unusually large amount of activity is desired. Most rail shipments will continue to be made with less than 15 mr/hr at the surface of the container to keep, insofar as possible, the same standards for air and rail shipments. As soon as regulations permit, air shipments will be made with higher than 15 mr/hr radiation at the surface. This will effect a considerable saving in weight and, consequently, in cost of air shipments.

The isotope clerical work, formerly handled by the Laboratory administrative organization, has been made a responsibility of the Operations Division.

[REDACTED]