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ORNL-27
Progress Report

OAK RIDGE NATIONAL LABORATORY

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OPERATING DIVISION REPORT

for

Month Ending March 31, 1948

PILE OPERATIONS
SEPARATIONS
RADIOISOTOPES

by

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

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ORNL-27
Progress Report

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SUMMARY

1. One ruptured slug, exposed for 107 days, was detected and removed without difficulty.
2. The excess reactivity of the pile is now about forty to fifty in-hours, a decrease of approximately twenty-five inhours.
3. Calcium nitrate slugs are being canned and loaded for C^{14} production.
4. The Operating Division suffered its first major injury since the start of the Laboratory in 1943.
5. RaLa Run #24, containing approximately 2,900 curies, was shipped on March 13, 1948, one week behind schedule.
6. Isotope production continued satisfactorily. Additional emphasis is being placed on fission product isolation processes.
7. The 299 shipments for this month sets a new record. The previous high month was January, 1948, with 223 shipments. A total of 2,940 shipments have been made since the start of the Program in August, 1946.

[redacted]

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A. 100 AREA OPERATION

I. Operating Data:

	March 1948	February 1948	Year to Date
Total Accumulated KWH	2,707,200	2,437,030	7,510,099
Average KWH/operating hour	3906.78	3790.63	3819.18
Average KWH/24-hour day	3638.71	3501.43	3440.02
Percent lost time	6.9%	7.6%	10.0%
Approx. Excess Pile Reactivity	40-50 inhours	70-75 inhours	
Slugs Charged	463	997	2493
Slugs Discharged	490	997	2586
Product Made (grams)	98.80	88.94	274.09
Product Discharged (grams)	4.62	22.46	53.70

II. Pile Operations:

One ruptured slug was found in the pile this month. This rupture occurred in metal channel 2165 on March 14, 1948. The slug, which contained a thermocouple had been in the pile for 107 days at a temperature of approximately 210°C. It was discharged without difficulty.

The wooden ends of all front face shielding plugs in thermocouple channels were changed this month. This completes the job for this period.

All metal channels which do not contain metal have been inspected and those which did not contain air blocks were closed with a lead slug and "spider". The pile differential pressure has been increased by two inches of water. The added cooling by the increased air flow over the metal has allowed the pile to run at a somewhat higher power level. Steady power runs have prevented the accurate evaluation of this increase.

Some difficulty was experienced with the trip chamber amplifiers. The pile was shut down several times due to the failure of these instruments. All equipment was overhauled and it is now functioning satisfactorily.

The pile operation was normal throughout the month with the usual shutdowns for removal and insertion of samples, discharge slugs for Building 706-D operations, and other miscellaneous work.

The excess pile reactivity is between forty and fifty inhours. This decrease of from twenty-five to thirty-five inhours during the month was caused by the insertion of two 900-gram antimony plaques and the loading of 1,495 calcium nitrate cans for C^{14} production.

III. Fan Operation:

All fans have operated without difficulty during the past month.

Both #2 and #3 fan control circuits have been cross-connected to the 220-volt plant electric system with a double-throw switch so that either the 220-volt control transformer in the fan cubicle, or the 220-volt plant electric system can be used to run the fan control circuits. This system has the further advantage of allowing the fan control circuit to be checked when the 2300-volt fan power circuit is off.

IV. Radioisotopes:

The following table is a record of the isotope and research samples charged into the pile during February and March, 1948:

	FEBRUARY		MARCH	
	Research	Radioisotopes	Research	Radioisotopes
Stringers 13, 14, and 16	10	95	49	108
Hole 22 (Pneumatic Tube)	65	0	59	0
All other holes	7	25	17	8
TOTAL BY GROUPS	82	120	125	116
TOTAL FOR MONTH	202		241	

As of March 31, 1948, there were 346 cans of target material in Stringers 13, 14, and 16, compared to 351 cans of target material at the end of February, 1948.

V. Miscellaneous:

The calcium nitrate canning program is progressing but not without difficulty. The welding machine used to weld the cans has required a large amount of maintenance work and, as yet, is not operating satisfactorily. It is felt, however, that these difficulties will be overcome shortly.

During the month twenty-three metal channels were charged with sixty-five calcium nitrate slugs each. This leaves twenty-five channels yet to be filled with calcium nitrate.

VI. Experimental Work:

The volume of experimental work in the Pile Area is increasing. A study has recently been started to determine the effect of neutrons on the concrete shielding. It is planned to take core drillings of the shield at various locations to determine the water content, compressive strength, etc., of the concrete.

An investigation was initiated within the past few days to determine the feasibility of cutting another large hole through the top shield to provide additional experimental facilities.

[redacted]

B. 706-D AREA

I. Barium (Ba^{140} - 12.5d)

Run #24 was shipped on March 13, 1948, one week behind schedule. Los Alamos reported this shipment contained approximately 2,920 curies, which agrees with our estimate.

The analytical summary of this run is tabulated below:

	Curies	Percent
Total Curies Dissolved	4276	100%
Cell A Losses	541	12.65%
Cell B Losses	248	5.80%
Total Accounted Losses	789	18.45%
Approximate Quantity Shipped	2920	68.33%
Material Balance		86.78%
Losses Unaccounted For		13.22%

The loss of time and product due to decay was again caused by incomplete metathesis. A thorough investigation of this condition revealed that it was caused by dilution of the K_2CO_3 concentration resulting from an excessive water heel left in the tank after the last extraction decantation. An increase in the concentration of the K_2CO_3 was tried on the second attempt to metathesize the product and the results were found to be satisfactory.

Following shipment of the run, Cell B and the cubicles were decontaminated to perform the following jobs:

1. Replace all Tygon lines in the cell with stainless steel pipe.
2. Replace the bearings on the product carrier dolly.
3. Replace the glass equipment and Tygon lines in the cubicles.

[redacted]

4. Repair the electrolysis tank which was found to have three leaks allowing the jacket cooling water to flow into the product solution when the pressure was greater than six pounds per square inch.
5. Replace black iron tank supports.
6. Check the cone heater thermocouple.
7. Perform any other maintenance work found to be necessary.

Run #25 is tentatively scheduled to start about the middle of May, 1948.

The study to use Hanford irradiated uranium in place of slugs exposed in the Oak Ridge pile is being continued. A preliminary design of a carrier for use in transporting slugs from Hanford to Oak Ridge by air has been sent to the General Electric Company for their comments and suggestions. If the design is satisfactory, one container will be built for test purposes.

SPECIAL NOTE: SAFETY

The first major injury to be sustained by an employee of the Operating Division since the start of the Laboratory in 1943, occurred during the month. An operator was burned about the face and neck with ninety percent sulphuric acid. After several days hospitalization, the employee returned to work. No permanent disfigurement nor injury resulted.

II. Radioisotopes:

1. Iodine (I^{131} - 8d)

Thirty 75-gram cans of irradiated tellurium were processed this month and approximately 3,399 millicuries of I^{131} were shipped. All of the product was within specifications.

Iodine Development Work

The dissolver cell in Building 706-C was decontaminated during the month to allow the installation of a drain line from the UNH transfer tanks to the sump. This will allow the tanks to be washed without sending the wash water to the fission product column cell.

A nitric acid coating-removal procedure, using mercuric nitrate catalyst, was tried on a batch of ninety-day slugs. The UNH solution contained 0.04 mg aluminum per ml, which is considered satisfactory.

Sintered stainless steel steam filters have been ordered to clean the steam used to sparge the dissolver solution. The steam sparging step is essential to remove the iodine from the dissolver solution, but any organic matter which may be introduced with the steam reduces the efficiency of the process.

Work continued on the installation of equipment in the iodine purification hood in Room 10, 706-C Building. Repair work in the main cell required a disproportionate amount of attention during the month, delaying work in Room 10.

[redacted]

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

Six 2000-gram cans of irradiated sulfur were processed. Approximately 2,943 millicuries of P^{32} were shipped.

Equipment was installed during the latter part of the month to improve the method of loading sulfur cans into the melter of the pressure process equipment. This new equipment is designed to reduce the radiation hazard. A run has not been made since the installation of this equipment.

Phosphorus Development Work

No further development work was done during the month.

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

Seven hundred $Ca(NO_3)_2$ slugs were processed in seven runs. Approximately 480 millicuries were produced with an isotopic ratio averaging 4.54% and ranging from 3.8% to 5.8%.

An attempt to increase the isotopic ratio was made by purging the equipment with nitrogen before processing the $Ca(NO_3)_2$ slugs. There was no noticeable improvement.

C^{14} Development Work - (From Be_3N_2)

Work continued on the installation of equipment in Building 204 Annex for the Be_3N_2-C^{14} process. The installation is about seventy percent completed.

4. Sulfur (S^{35} - 87.1d)

Approximately 650 millicuries of S^{35} as sulfate were prepared from irradiated KCl during the month.

[redacted]

5. Fission Products

One run was finished and another is now in progress on the all-column fission product equipment. Approximately five curies of fission product activities were obtained in the various fractions, which were removed for purification on apparatus outside the cell.

Zr-Cb fractions are being purified by extracting the Zr and alpha emitters into TTA-benzene. Alpha activity is removed with dilute acid and the Cb is removed as it grows in by extracting with strong HCl. Various methods are being tried at present to remove the Zr-Cb mixtures in a medium suitable for shipment. All outstanding orders for Cb were filled during the month.

Yttrium continued to be produced in small amounts, twenty-five to fifty millicuries per week, which is being used to fill outstanding orders. The method has been improved by eliminating a TTA-benzene extraction step which was used to remove alpha emitters; the purification is now handled entirely on columns by careful adjustment of pH and use of ammonia from Amberlite resin.

Small quantities of fairly pure rare-earth fractions, low in cerium and yttrium, have been obtained by fractionation on resin columns. The alpha contamination has been reduced to a sufficiently low level so that the material may be shipped to fill some outstanding orders. These rare-earth fractions have not yet been further separated to yield pure Pr or Nd.

No attempt is being made at the present time to purify the fractions containing some of the other fission products, such as Ba, Sr, Ce, Cs, and Ru.

6. Ruthenium (Ru^{106} - ly)

The extensive changes being made to the precipitation equipment in the Tank Farm Area are almost completed. Heavy shielding was added to increase protection from radiation, remote controls installed, and a storage tank was provided to increase the capacity of the plant. This equipment will be operating during the coming month and should process a large amount of the W-7 supernatant liquor.

The purification of Ru by distillation from the iron hydroxide precipitate on which it is carried has been improved considerably so that yields of seventy to eighty percent are obtained consistently. The ferrous hydroxide precipitate is dewatered as much as possible by allowing it to stand and decanting the supernate carefully. The precipitate is dissolved in sulfuric acid and passed to a concentrating still where more water is removed and some of the traces of oil present is steam distilled out. Air is bubbled through at this point to oxidize the ferrous iron; nitric acid is added to promote the oxidation. The solution is transferred to another still, more sulfuric acid and potassium permanganate are added, and the ruthenium is distilled as the oxide into HCl, where it is reduced and trapped. A number of distillations are made into the same receiver, after which the HCl is distilled off. Considerable inactive Ru formed

in fission makes the specific activity fairly low, about one millicurie Ru^{106}/ milligram Ru. Approximately 110 millicuries of Ru^{106} were produced during the month to fill outstanding orders.

7. Calcium (Ca^{45} - 180d)

About fifty millicuries of Ca^{45} were purified from Hanford-irradiated material during the month. Six side hole tubes, each containing ten grams $CaCO_3$, were prepared and sent to Hanford for irradiation.

Purification of scandium and packing in cadmium-shielded tubes for irradiation in the Hanford Pile continued.

8. Strontium (Sr^{89-90} - 55d, 30y)

One run was made from approximately fifty milliliters of HCl-ether waste from Ba^{140}, Run #23. The run yielded about 2,140 millicuries of Sr^{89-90} with approximately ninety-five percent of it being Sr^{89}.

9. Iron (Fe^{55-59} - 44d, 4y)

No further work this month.

10. UX_1 (Th^{234} - 24.5d)

1.35 millicuries of UX_1 were prepared from K-25 residue for a special order.

11. Zinc (Zn^{65} - 250d)

Work was resumed on the separation of Zn from copper cyclotron target backings which had been bombarded with deuterons. However, only 0.1 millicurie of Zn^{65} is obtained per backing, which makes production of Zn^{65} impractical unless we can

obtain specially-bombarded copper targets. The specific activity of the preparation was 0.3 millicurie Zn^{65}/ milligram Zn and the purity was ninety-nine percent.

No further work on this problem is planned.

12. Miscellaneous Materials

Samples of antimony, selenium, cadmium, mercuric oxide, thallous nitrate, and tungsten oxide were prepared in side-hole tubes for Hanford irradiation.

Attempts to dissolve an irradiated sample of tantalum oxide in molten KOH were not completely successful and some method of improving this procedure is being sought.

13. Organic Synthesis

Two shipments of C^{14} labeled methanol (3.04 mc) were made during the month from material prepared under the direction of the Chemistry Division.

III. Tank Farm and Burial Ground:

1. Tank Farm

(a) The program to provide space in the metal waste tank (M-3) for the aluminum-plutonium waste from the Hot Pilot Plant was essentially complete at the end of the month with only two inches of uranium slurry remaining.

It is doubtful that further attempts to make a complete transfer will meet with success.

(b) With the exception of the installation of an automatic jet and float, the new chemical waste diversion tank, W-12, project has been completed. This tank is now being used to replace the old gunite tank (W-11).

(c) A total of sixteen, fifty-liter pots and eleven, sixty-five-gallon drums were received from Chicago this month. All of the pots contained fission products only. Six of the drums contained 113.8 kilograms of uranium; the remaining five contained aqueous solutions for disposal.

(d) The Hot Pilot Plant transferred 138.22 kilograms of uranium to W-3 metal waste tank.

(e) The program to replace the poles supporting the steam lines in the Tank Farm was continued this month. It is about seventy-five percent completed.

(f) In order to make more room for metal waste, a new jet line was installed from W-9 to W-8 tanks. A total of 68,400 gallons of metal waste was transferred to W-8 from W-9. We now have enough free space in W-9 for the dissolved metal from approximately ten barium runs.

(g) The 706-D Operation transferred 6,279 gallons of metal waste from Barium Run #24 to W-9 tank.

(h) The following is a listing of the movement of liquids in the Tank Farm for March, 1948:

WATER WASTES

Tanks	Capacity	Est. Amt. Rec'd-Mar.	Disposed Of	Discharged To	Free Space
W-1 & 2	8,800 gal.	214,370 gal.	214,370 gal.	Settling Basin and W-5	8,800 gal.

About fifteen percent of this waste was sent to W-5 tank and the remainder went to the Settling Basin.

CHEMICAL WASTES

W-5 & 6	280,000 gal.	140,400 gal.	123,600 gal.	Settling Basin	105,000 gal.

Approximately eighty-five percent of the chemical waste received originated at 706-A, C, and D. The remainder came from W-1 and W-2 tanks.

METAL WASTES

W-3-4-7 -8-9-10	755,700 gal.	9,426 gal.	0	----	168,334 gal.

Approximately sixty-seven percent of the metal waste came from 706-D Building. The remainder came from the Hot Pilot Plant and dilution from steam in jetting.

SETTLING BASIN

Total Est. Discharge	Total Curies Discharged	Beta counts/min/ml Average	High	Low	Discharged To
28,292,000 gals.	91.93	253.6	1041	59	White Oak Creek

RETENTION POND

467,129 gals.	.141	23.4	226	1	White Oak Creek

2. Burial Grounds

Besides routine burials of contaminated materials from the Restricted Area, four shipments of alpha contaminated waste from Dayton, one load of rags from Chicago, and two carboys of waste from Y-12 were buried.

[redacted]

C. RADIOISOTOPE PRODUCTION AND SHIPMENTS

General:

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

	FEBRUARY 1948	MARCH 1948	TOTAL AUGUST, 1946, to MARCH, 1948, Inc.
Separated Material 706-D Area	152	221	2,090
Unseparated Material 100 Area	51	78	850
TOTAL	203	299	2,940

Included in the above figures are eight shipments during February and twenty-three during March to institutions in foreign countries.

The two hundred ninety-nine shipments this month represent an increase of forty-seven percent over February and an increase of thirty-four percent over the previously high of two hundred twenty-three shipments for January, 1948.

[redacted]

[redacted]