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Progress Report
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OAK RIDGE NATIONAL LABORATORY

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OPERATIONS DIVISION REPORT

for

Month Ending May 31, 1948

PILE DEPARTMENT
CHEMICAL SEPARATIONS DEPARTMENT
RADIOISOTOPE DEVELOPMENT DEPARTMENT
ISOTOPE CONTROL DEPARTMENT

by

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SUMMARY

1. Two slugs with ruptured jackets were detected and discharged.
2. The excess pile reactivity is only about twenty inhours. Additional metal may have to be added to provide for several proposed experiments.
3. The canning of $Ca(NO_3)_2$ for C^{14} production has been completed.
4. The bearings on the #3 fan were replaced during the month. During this period the pile operated at half power.
5. Isotope production continued with minor difficulties experienced in the operation of the P^{32} isolation equipment.
6. The fission product column cell has been decontaminated and new equipment is being fabricated.
7. RaLa run #25 has again been postponed by Los Alamos. It is now scheduled to start about the first of July, 1948.
8. The problems of transporting and using Hanford slugs for RaLa production are being investigated.
9. There were 281 isotope shipments this month as compared with 269 in April, 1948. The weekly average for May, 1948, is the highest to date and in the last week of May there were the largest number of shipments, seventy-eight, that we have had so far.
10. The Source and Fissionable Material Accountability work was transferred from the Security and Protection Department to the Isotope Control Department of the Operations Division.

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A. PILE DEPARTMENT

I. Operating Data:

	May 1948	April 1948	Year to Date
Total Accumulated KWH	2,549,649	2,541,495	12,601,243
Average KWH/operating hour	3686.91	3693.34	3761.31
Average KWH/24-hour day	3426.95	3529.85	3454.29
Percent Lost Time	7.0%	4.3%	8.3%
Approx. Excess Pile Reactivity	20-30 inhours	60-70 inhours	
Slugs Charged	89	11	2593
Slugs Discharged	167	11	2761
Product Made (grams)	93.05	92.76	459.90
Product Discharged (grams)	7.20	0.03	60.93

II. Pile Operation:

Slugs with ruptured jackets were detected on two different occasions during the month, as follows:

Date	Channel No.	Exposure Days	Temp. Range	Slugs in Row
May 2, 1948	2170	110 days	235 - 245°C.	36
May 9, 1948	1159	709 days	150 - 160°C.	50

The normal uranium slugs were discharged from two channels (1768 and 1968) and replaced with uranium "doughnuts" to provide additional facilities for fast neutron experiments. Each of these holes now contains seven "doughnuts" located about the middle of the pile. An aluminum tube extends from the front face back through the doughnuts to facilitate the insertion and removal of samples. These installations are similar to the other fast neutron hole, Channel 1867, which has been in use since 1944.

The removal of the slugs from the above mentioned channels, the warmer weather, and the insertion of miscellaneous materials reduced the available excess reactivity to practically zero. By draining the

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water from the tube in Hole 19 a sufficient number of inhours were realized to maintain proper operating conditions. The installation of various pieces of experimental equipment within the next few months will require approximately one hundred inhours. The problem of increasing the available excess reactivity is being studied by personnel from the Physics Division.

The eleven slugs loaded into Channel 1964 in April, 1948, continue to operate satisfactorily at temperatures between 250 - 350° C. This test is being run by the Physics Division in an attempt to avaluate the aluminum-jacket life at temperatures higher than the present 250°C. level.

With the exception of the inhour shortage and the two ruptured alug jackets the pile operation was normal for the past month. The warmer weather and the shutdowns for scanning account for the lower power level and the increased lost time.

III. Fan Operation:

The bearings on the #3 fan were replaced on May 4, 1948. The average life of bearings has been six to eight months. These had been in service for thirteen months. No explanation can be given for the apparent improvement. The fan was decontaminated and the bearings changed with no unusual difficulties. The outboard bearing was in poor condition. Approximately half of the balls were badly pitted.

The #2 fan operated normally throughout the month.

IV. Radioisotopes:

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

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	APRIL		MAY	
	Research	Radioisotopes	Research	Radioisotopes
Stringers 14, 14, and 16	29	102	3	119
Hole 22 (Pneumatic Tube)	35	0	41	0
All Other Holes	13	10	18	8
TOTAL BY GROUPS	77	112	62	127
TOTAL FOR MONTH	189		189	

At the end of May, 1948, there were 365 cans of target material in Stringers 13, 14, and 16, compared to 392 cans of material in these stringers at the end of April, 1948.

V. Miscellaneous:

The calcium nitrate canning program has been completed. There are now 6,529 cans of calcium nitrate in the pile with 1,344 cans in stock for replacements. It is planned to continue the exposure of $Ca(NO_3)_2$ to insure the C^{14} production until a satisfactory process can be developed for isolating the C^{14} from Hanford exposed Be_3N_2.

VI. Experimental Work:

Arrangements are being completed for the procurement of the equipment required for taking core samples from the pile shielding. These samples are to be used to determine the effect of several years' exposure to neutrons on the structural characteristics of the concrete.

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B. CHEMICAL SEPARATIONS DEPARTMENT

I. Radioisotopes:

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

Forty-two, seventy-five-gram, cans of irradiated tellurium were processed and approximately 4,594 millicuries were shipped. All product was within specifications.

Fission Product Iodine Development

One run was made during the month for the Chemistry Division. This was a special run in which the slugs were removed from the pile, dissolved, and the iodine recovered within twenty-four hours to secure short-lived material for research work.

Two routine inspections of the cell equipment were made and several leaks discovered and repaired. A large screen was installed over the sump to prevent clogging. A draw-off line was installed in the catch tank so that special research samples may be drawn off when needed.

All equipment has been installed in the purification hood in Room 10. Water test runs are being made. The equipment shielding was tested with a 200-mc Co^{60} source. A reading of thirty mr/hr was noted through the three-inch lead shielding on contact at a point directly opposite from the source. Particular attention was paid to scattering; no areas were found with high radiation due to scattering. A minitron, seven feet above the floor and five feet from the vertical face of the shield indicated two mr/hr. This test indicates that the shield will be adequate, since cobalt has more energetic gamma radiation than iodine.

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

Twelve, two-thousand-gram, cans of irradiated sulfur were processed. Approximately 3,428 millicuries of P^{32} were shipped.

Plugging of equipment during several of the runs resulted in low yields. An investigation indicated that the operating temperature of the equipment was too high. Subsequent runs, made with a revised operating procedure with a ten-degree lower temperature, gave no difficulty.

Phosphorus Development Work

Some work is being done on the basic chemistry and physics of the separation of P^{32} from irradiated sulfur to furnish data for a better understanding of the difficulties experienced with the present nitric acid process and to design a new procedure. A literature survey is being made and initial experimental work is being directed toward determining the form in which phosphorus occurs in irradiated sulfur.

It has been noted that thirty to forty percent of the P^{32} may be removed from the molten sulfur by passing a stream of carbon dioxide through it. Past work has also indicated that some P^{32} may be present in a volatile form.

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

No runs were made.

Beryllium Nitride Development Work

Most of the large equipment , such as furnaces, mantle heaters, and rheostats have been received. Two important parts are not

yet here: the Brown recording potentiometer and the Zeus-type amplifier. Equipment is being installed in the melting and crushing hood and development work will start at this point in the next few weeks.

There is some indication that carbon dioxide is absorbed in beryllium nitride or that it contains carbonates from the original manufacturing process. Since this would be very bad for the C^{14} process because it would reduce the specific activity, an assay was made on a sample of crushed beryllium nitride that had been standing in the open for six months. About 165 micrograms of carbon were obtained per gram of beryllium nitride. This indicated that CO_2 contamination is quite appreciable and will have to be eliminated if high specific activities are to be obtained. There is some possibility that the inactive carbonate will be decomposed in the furnace during melting of the jacket. The fate of active and inactive carbon during the furnace treatment will be one of the first development problems studied.

4. Sulfur (S^{35} - 87d)

A thirty-four-gram sample of Hanford-irradiated KCl was processed to produce approximately 1,250 millicuries of Na_2S^{35}. Various customers have indicated that our sulfide apparently is oxidized to some extent. We confirmed this by our own tests and are at the present time searching for a method of preventing the oxidation. Initial tests with organic reducing agents such as hydroxyl amine and hydrazine were not successful. If solutions of alkali sulfides prove too unstable for shipment, it will be

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necessary to ship precipitated metallic sulfides, hydrogen sulfide gas, or perhaps discontinue the production of S^{35} as sulfide.

5. Fission Products

Work was continued on the decontamination and removal of old equipment from the column cell. This phase of the remodeling of the fission product column equipment is now complete. The cell background is about twenty mr/hr. New equipment is being designed and will be fabricated as fast as conditions in the shops allow. Some extra lead shielding will be installed at points where experience has shown it is needed.

A number of new ion exchange resins have been received and these have been turned over to the Chemistry Division for evaluation.

If space allows, provision will be made for making solvent extractions in the cell to simplify removal of Pu.

New remote control pinch clamps for Tygon tubing have been designed to eliminate glass stop cocks or metal valves wherever possible.

Six lead-shielded columns have been set up on the east side of 706-D. These columns extend from the third floor to the first floor and will contain resin columns in Tygon tubing up to eighteen feet in length with a total head of about thirty feet. Most of the resin beds will probably not be over six feet in length. In addition to these columns, two small lead-enclosed columns have been installed for changing from citrate to HCl solution and con-

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centration of final products.

Approximately one hundred millicuries of yttrium was purified during the month by a combination TTA extraction and resin column procedure. The product contained less than seventy alpha counts per millicurie of yttrium.

Rare earth citrate cuts from the all-column equipment were converted to HCl solution by column technique and concentrated to a small volume by evaporation. This material was greatly diluted, pH adjusted to 2.0, and the activity placed on an eighteen-foot IR-100 column. The band of activity was only two inches wide, which is an ideal condition for fractionation.

The activity was eluted from the column with 2.75 pH citrate at a rate of one column volume per hour. The elution required 435 hours. A summary of the run is as follows: the "Y" peak occurred in the first thirty hours; the alpha activity was removed during the first sixty hours; from the 130th hour to the 170th hour pure praesodymium was eluted; after 230 hours pure cerium was eluted; activities left on the column after 435 hours were removed in 6N HCl and identified as mainly Ba, Sr, and Ce.

About forty millicuries of Pr was obtained which was shipped on some long-standing orders.

The Zr-Cb purification apparatus was put into operation during the month. About 170 millicuries of Zr-Cb was purified in the one run made. The alpha count was reduced to less than 200/mc of Zr-Cb. This product contained about four percent Cb. Total solids were

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3.8 mg/ml and the spectrographic analysis showed no unusual contamination. This product is being readied for shipment. The columbium that was extracted during the process will be further purified and held in stock.

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

Sixty-two ruthenium concentrates have been made, using approximately 37,200 gallons of supernatant liquor from W-7 metal waste tank.

The yields have varied from time to time but special sampling has indicated that the position of the remotely operated sampler in the tank has caused false results and that actually all yields are uniformly high at about sixty percent.

Ruthenium Development Work

Work continued on the re-carrying of Ru from iron solution on CuS. For this procedure it is necessary to keep the iron in the reduced state and for this purpose sulfur dioxide gas is being used. The pH is adjusted with caustic soda and carbon dioxide gas is bubbled through the solution to remove excess SO_2. Copper sulfate is added, the solution made to five percent sulfuric acid strength, and hydrogen sulfide bubbled through to precipitate CuS. A considerable amount of elemental sulfur comes down as well as the sulfide, but it has been found that the sulfur carries Ru just about as well as CuS. The precipitate is settled and the liquor drawn off. The sludge is removed for the Ru distillation process. About ninety-eight percent of the Ru is carried in this process. Use

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of this re-carrying procedure should reduce the number of Ru distillations considerably.

Some progress has been made in the installation of Ru distillation equipment for routine production work on the third floor of 706-D. Progress on this installation depends upon the rate at which construction work can be done by the Mechanical Department.

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

Equipment for the separation of Ca^{45} from scandium has been completed and tested. This project will go forward as soon as personnel can be assigned to it.

An adequate supply of high specific activity Ca^{45} solution made from Hanford-irradiated material is on hand.

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

One run was made. This run yielded approximately 4,000 millicuries of strontium which analyzed 59.8 percent Sr^{90}.

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

A fourteen-gram, Hanford-irradiated, iron sample was processed. The yield was thirty-five millicuries Fe^{59} by gross beta count with a specific activity of 0.0026 mc/mg Fe. Fe^{55} is being counted on a new counter developed by the Chemistry Division and the results are not yet available.

10. Miscellaneous Work

Processing work was completed on one unit each of Sb^{125} and $Hg^{203-205}$. These materials were shipped.

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Some special work was done on the separation of yttrium and element 61.

Building materials submitted for examination were given tests to determine relative ease of decontamination. Iodine was used as the test activity.

II. Tank Farm and Burial Ground:

1. Special Wastes

Other than routine disposal of plant wastes, the following special wastes were handled:

a. Twenty-three pots and five drums were received from Chicago. All the pots contained fission products only. One of the drums contained 18.49 kilograms of uranium; the others hexone and fission material.

b. Four alpha-contaminated shipments from Dayton were buried at the west burial grounds.

c. The ruthenium process consumed 27,400 gallons of precipitated metal supernatant which contained an average of .0035 percent uranium.

d. The Hot Pilot Plant transferred 33,85 kilograms of uranium to W-4 and 8.37 grams of plutonium to W-3.

e. The 706-D operation transferred 1,624 gallons of metal waste to W-9 tank.

f. Approximately 26,864 gallons of precipitated metal supernatant were transferred from W-4 tank to the chemical waste system. This liquid contained .012 percent uranium.

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g. Attempts were made to remove algae from the Settling Basin. Approximately 150 gallons of formaldehyde were added and the Basin was also hosed down twice with a fire hose. Both methods removed the algae temporarily.

2. Wastes Discharged to the White Oak Creek

The east and west ponds were used this month to discharge activity to the creek. This was done when a large amount of water leaked into W-5 tank, overtaxing the chemical waste system to the point where it was impossible to continue discharge of wastes through the Settling Basin and still stay under the five-curies-per-day tolerance limits. The leak to W-5 was found to be from a raw water plugcock which was discharging underground, draining into the W-5 diversion box. After this leak was stopped, the project to replace all woodwork at the Settling Basin was started and the east and west ponds were continued to be used to prevent any activity from entering the Basin while this work is proceeding.

a. From Settling Basin

Gallons Discharged	To	Curies
20,415,000	White Oak Creek	79.25

b. From East and West Ponds

Gallons Discharged	To	Curies
1,581,000	White Oak Creek	32.96

c. The Retention Pond discharged 232,000 gallons of water carrying .12 curie of beta activity.

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3. Waste Tank Inventory

The following is the inventory of the waste tanks as of May 31, 1948:

Tank	Gallons Capacity	Gallons In	Gallons Out	Discharged To	Free Space
			CHEMICAL WASTE		
W-1-2-5-6	348,800	374,500	442,900	Settling Basin	226,000
			METAL WASTE		
W-4-7-8-9-10	713,000	2,808	52,264	Chemical Waste System	180,560
			Al-Pu WASTE		
W-3	41,300	Gauge being repaired.			

4. Maintenance

a. A project was begun at the end of the month to replace all of the above-ground transfer lines in the Tank Farm. This new line will be permanent and constructed for efficient operation.

b. The project to replace all the catwalks, weirs, and baffles in the Settling Basin was begun and is now about twenty-five percent complete.

c. The leaks in the W-6 to W-9 jet line, which were reported last month, were repaired by installing new gaskets in the flanges located in the jet pit.

d. All new float gauges have been installed in the waste tanks in the Tank Farm. W-1, 2, 3, and 4 remain to be calibrated.

e. The pole replacement program in the Tank Farm has been completed.

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f. A small CL #2 jet was installed, jetting from W-6 to the Settling Basin. This jet was installed to facilitate the removal of waste from W-6 to the Basin in a steady stream. This jet delivers three and one-half gallons per minute.

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III. RaLa (Barium - Ba^{140} - 12.5d):

All repairs in Cell B (706-D Building) have been completed and the equipment checked. Los Alamos again postponed run #25 until the first part of July, 1948.

During the equipment testing in Cell B, the jet used to empty the cell ventilation ducts failed. A new line was installed through a Cell B louvre. The original line was abandoned until Cell A can be decontaminated sometime in the future.

Fabrication of the slug carrier for the transportation of Hanford slugs to Oak Ridge is about seventy-five percent complete. Several tests to determine the heat dissipation of the carrier are planned.

Three "dummy" runs, using Hanford slugs, were made during the past month to determine the process changes necessary. These runs revealed that the following modifications must be made:

1. Two-coating-removal treatments instead of the usual one will be necessary to remove all of the aluminum jacket.
2. The coating removal digestion time will be increased from forty-five minutes to four hours.
3. Six water washes, instead of the present two, will be required to remove all of the aluminum salts and bonding materials.
4. The dissolving time will be increased from about six hours to approximately twenty hours.

As soon as the transportation container can be completed and tested, a formal proposal to use Hanford slugs for the production of RaLa will be made.

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C. ISOTOPE CONTROL DEPARTMENT

I. General:

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

	April 1948	May 1948	Total August, 1946, to May, 1948, Inc.
Separated Material			
706-D Area	211	222	2,523
Unseparated Material			
100 Area	58	59	967
TOTAL	269	281	3,490

The 281 isotope shipments this month represents the highest number of shipments to date based on the number of weeks in May. In March there were 299 shipments but this was because there were effectively five weeks in March.

II. Iodine Distribution:

There has been a fifteen percent increase in the amount of iodine distributed in the past month. Production has been slightly behind demand at certain times and apparently this condition will continue until the fission iodine becomes available. The interest in the use of iodine is probably due to the success which has been encountered in the treatment of thyroid cancer with this isotope.

III. High Specific Activity Iron:

A shipment of iron was obtained from Hanford with a specific activity of about five millicuries per gram. There is a considerable

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III. High Specific Activity Iron: - Continued

demand for this material for use in biological experiments where a large amount of iron cannot be injected into the test organisms. This specific activity compares with .06 millicurie per gram obtainable in the ordinary irradiation unit which we produce in the Oak Ridge National Laboratory pile.

IV. Other High Specific Activity Units:

There is a widespread demand for high specific activity isotopes of such elements as zinc, calcium, cobalt, and others, which cannot be met because of the low flux available in the Oak Ridge National Laboratory pile. The only pile with a higher flux available is at Hanford and a large number of irradiations are being made there for this purpose.

A large amount of interest in radioactive cobalt as a substitute for radium in medical therapy has been generated by the recent Atomic Energy Commission publicity release. We now have tentative orders for about 600 curies of this material. These orders are so large that production will have to be spread over several months and, as the demand continues to grow, other facilities will probably be required.

V. Fission Products:

Several of the long outstanding orders for Pr^{143} have been shipped this month. Zirconium and columbium are expected to be available by the first week of June, 1948.

VI. Pile Flux:

The recent declassification of the pile flux will enable us to furnish customers the intergrated flux received by their irradiation units when requested. [redacted]

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VII. Delay of Shipments by Threatened Rail Strike:

In the first week of May, the threatened rail strike caused the delay of several shipments scheduled to go by rail. Fortunately, the strike did not materialize and shipments were sent a week later.

VIII. Source and Fissionable Material Accountability:

The Accountability Representative inventoried the uranium storage vault (Building 103) and assumed responsibility for the Source and Fissionable material stored therein. In the future, material will be issued by the Accountability Representative as required.

Accountability Reports for the month of April were submitted to the Atomic Energy Commission under date of May 14, 1948. This represents a continued reduction in the time lag required in compiling plant-wide inventories and preparing Material Balance Accountability Reports and is the first time in the history of the Laboratory that reports have been submitted on time as scheduled.

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