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ORNL-85
Progress Report

OAK RIDGE NATIONAL LABORATORY

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OPERATIONS DIVISION REPORT

for

Month Ending June 30, 1948

PILE DEPARTMENT
CHEMICAL SEPARATIONS DEPARTMENT
RADIOISOTOPE DEVELOPMENT DEPARTMENT
ISOTOPE CONTROL DEPARTMENT

by

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SUMMARY

1. The pile loading was increased from 48.5 to 52.1 tons of uranium. A gain of approximately 160 inhours was realized.

2. A minor fire in a 2300-230-volt transformer in the starter cubicle of the #3 fan occurred during an electrical storm.

3. A slow neutron animal exposure chamber was installed in the thermal column on top of the pile.

4. Isotope production continued normally throughout the month.

5. The supernatant liquid on top of the precipitated uranium in tank W-7 has been processed through the Ru^{106} isolation equipment. The material from tank W-10 will next be processed.

6. RaLa Run #25 has again been postponed by Los Alamos. It is now scheduled to start on July 12, 1948.

7. There were 327 isotope shipments for the month of June 23rd compared with 281 in May, 1948. In the week ending June 11th, there were the largest number of shipments (eighty-two) in any week since the start of the Isotope Program and the total of the month is also the largest of any month to date.

8. Since considerable interest has been shown in very large orders of Co^{60} and Ta^{182}, arrangements are being made to irradiate cobalt at Hanford. Also, special cobalt needles will be irradiated at Hanford to provide Co^{60} activity in a form comparable to small radium needles. These cobalt needles may be used in place of radium needles in standard radiological equipment.

SUMMARY - Continued

9. Orders for sulfur 35 in the form of sodium sulfide are being held pending development of a means of preventing oxidation of the sulfide.

10. Arrangements are being made to distribute C^{14} labeled compounds produced at the University of California. According to present plans, the material will be packaged in standard-size samples at the University of California and sold by the Oak Ridge National Laboratory under allocation by the Atomic Energy Commission.

11. Starting July 1, 1948, a cost accounting system will be put into effect which will give separate cost figures on I^{131}, P^{32}, and C^{14}. The balance of isotope costs will be accumulated for irradiated units and for miscellaneous processed isotopes.

12. The number of new orders received in June was the largest of any month so far. There were 125 non-project, fifty-five project, and twenty-two foreign orders.

A. PILE DEPARTMENT

I. Operating Data:

	June 1948	May 1948	Year-to-Date
Total Accumulated KWH	2,392,737	2,549,649	14,993,980
Average KWH/operating hour	3670.52	3686.91	3751.38
Average KWH/24-hour day	3323.25	3426.95	3433.35
Percent Lost Time	9.5%	7.0%	8.5%
Approx. Excess Pile Reactivity	190-200 inhours	20-30 inhours	
Slugs Charged	2813	89	5406
Slugs Discharged	64	167	2825
Product Made (grams)	87.33	93.05	547.23
Product Discharged (grams)	0.85	7.20	61.76

II. Pile Operation:

Due to the growth of the Radioisotope Program and in order to gain sufficient reactivity to permit the installation of additional experimental equipment, the pile loading was increased from 48.5 to 52.1 tons of uranium. The additional slugs were loaded only into the channels that already contained slugs. Since the charging was done in proportion to the amount of metal already in the channel, three slugs were added to each thirty-six, forty, and forty-four-slug row with four slugs being added to the fifty-slug channels. The loading of the additional metal has increased the pile excess reactivity by about one hundred and sixty-five inhours. This gain is considerably greater than was anticipated.

The nineteen rows of slugs, which were moved in April to evaluate their effect on the slow neutron flux in the animal tunnels, were returned to their former positions. This eliminates the distorted flux pattern in the upper regions of the loading.

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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 to evaluate aluminum slug jackets at temperatures above the present 250°C. level.

There have been no cases of ruptured slug jackets this month.

The pile-down time is higher than usual this month due to the pile being down thirty-two hours for the installation of an exposure chamber in the thermal column for the Biology Division.

III. Fan Operation:

Both fans have operated normally throughout the month except for minor difficulties in the auxiliary equipment.

A bearing burned out on the No. 2 oil pump on the No. 3 fan bearing lubrication system on June 18, 1948. The pump was replaced with a spare unit.

The 220-volt holding transformer on the No. 3 fan control system burned out during an electrical storm on June 27, 1948. The small fire which started was put out with no further damage other than the loss of the transformer. A spare transformer which had been installed to facilitate the checking of the No. 3 fan control system was put in service with the fan being out of service about three hours.

IV. Radioisotopes:

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

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	MAY, 1948		JUNE, 1948	
	Research	Radioisotopes	Research	Radioisotopes
Stringers 13, 14, and 16	3	119	53	126
Hole 22 (Pneumatic Tube)	41	0	27	3
All Other Holes	18	8	15	10
TOTAL BY GROUPS	62	127	95	139
TOTAL FOR MONTH	189		234	

At the end of June, 1948, there were 368 cans of target material in Stringers 13, 14, and 16, as compared to 365 cans of material in these stringers at the end of May, 1948.

V. Experimental Work:

A core was taken through the rear wall of the pile during June and at least one more core is to be taken through the pile walls during July. These core samples are to be used to determine the effect of several years' bombardment by neutrons on the structural characteristics of the concrete.

The thermal column was dismantled and rebuilt including a bismuth chamber to be used for slow neutron exposures by the Biology Division. The chamber has walls about eight centimeters thick and inside dimensions of approximately 6" X 6" X 8" and is located in the center of the thermal column about two and one-half feet above the pile graphite. The chamber is equipped with an access channel for insertion and removal of small animals or samples.

B. CHEMICAL SEPARATIONS AND ISOTOPE DEVELOPMENT DEPARTMENTS

I. Radioisotopes:

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

Forty-eight, seventy-five-gram,cans of irradiated tellurium were processed and approximately 5,127 millicuries of I^{131} were shipped. All of the product was within specifications.

Fission Product Iodine Development

Three dissolvings were made during the month for iodine development work. Two experimental runs were made for the Chemistry Division to produce short-lived iodine for research work.

The nitric acid (mercury catalyst) aluminum coating removal procedure was used in all runs. Indications are that serious mercury contamination of the UNH solution occurs which interferes with the volatilization of iodine. Efforts to completely eliminate mercury contamination were unsuccessful. It will probably be necessary to use an alkaline coating removal procedure, similar to that used in our initial work. Use of caustic solutions was previously discontinued because of corrosion to the tantalum lining; however, low concentration of caustic may be used on tantalum with a very low corrosion rate. Experiments indicate that approximately five percent NaOH solution may be used with comparative safety.

Tests were completed on the I^{131} purification equipment in Room 10. A purification run was made on a 200-mc batch of crude

iodine distillate from the cell. Nitrates were successfully removed by reduction to ammonia with ferrous hydroxide and subsequent distillation. Product iodine was recovered by distillation from the residue, which was adjusted to twenty percent H_2SO_4 and treated with ferric-ferrous sulfate as an oxidizing agent. A 6:1 ferric/ferrous ratio was used. The overall recovery from the crude distillate to final product was eighty percent. The product was of satisfactory purity.

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

Eight 2000-gram cans of irradiated sulfur were processed and approximately 3,678 millicuries were shipped.

One transfer line continued to plug during the runs made this month in spite of the lowered operating temperature mentioned in last month's report. It now appears to be caused by an uneven heating of some of the equipment. To correct this condition some equipment alterations are planned for the coming month.

Phosphorus Development Work

Small-scale glass apparatus is being used in continuing our investigation of the acetic acid extraction process. The equipment is designed so that volatile compounds of phosphorus and sulfur may be captured and studied. No experimental data are available at this time.

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

No runs were made.

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Beryllium Nitride Development Work

The furnace to be used for melting aluminum jackets and lead weights away from the Be_3N_2 pellets was tested. The jacket removal was very satisfactory. The apparatus has been designed so that an atmosphere of nitrogen is maintained in the furnace during the melting process. The gas is slowly drawn out of the furnace for a study of the volatile products formed during the melting operation.

Electronic equipment needed for the rest of the process has not yet been received.

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

Tests were made on various methods of maintaining S^{35} in the sulfide state in our dilute solutions used for shipment. The best method discovered so far is scrubbing H_2S^{35} from the process in barium hydorxide solution and storing this under a nitrogen atmosphere. The barium hydroxide solution is prepared oxygen-free and filtered before use. Storage stability tests have been underway for two weeks. During this time less than ten percent of the sulfide has been converted to a form other than sulfide, as determined by precipitation as cadmium sulfide.

5. Fission Products

The fission-product column cell was painted, lead installed around receiving and holding tanks, and some of the equipment installed. Twenty-six working drawings were made; sixteen work orders and six purchase requisitions issued. One special piece of equipment was made of fluorethylene plastic at K-25. If all

equipment is received on schedule, the installation should be completed during the coming month.

Four, six-foot, ion-exchange columns are in operation in 706-D. These individually-lead-shielded plastic tube columns are very satisfactory. An experimental run was made on one with UNH solution from Hanford slugs, using solution from the pilot plant dissolver. After U removal, all the other activities were removed with HCl; the HCl removed by evaporation and the diluted solution placed on another column. This simulates the technique to be used in the main fission product cell. The trial run was successful, indicating that our new design is basically sound.

Several column methods were tried for Zr-Cb purification. Activated carbon, silica, and anion exchange resin did not look promising. A new technique, using cation-exchange resin, and selectively removing Zr-Cb and Pu with oxalic acid adjusted to a pH of 1.0. A separation method using 2.75 pH citrate was also tried for the same purpose. Both methods show promise. About forty millicuries of Zr-Cb with $>$ twenty alpha counts per millicurie Zr-Cb was prepared for shipment. Sixty millicuries of Zr-Cb with a higher alpha count was also prepared and will be used for shipment to an Atomic Energy Commission location.

Approximately seventy-five millicuries of Cb, free from Zr, was prepared. The alpha count is $\geq$ 150 per millicurie Cb.

All stock solutions are being analyzed in order to prepare an inventory of material we have on hand.

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

Tank W-7 has been almost emptied of supernatant liquor. The ferrous hydroxide slurry accumulated in the storage tank will be re-processed to reduce volume. Chemical conditions in the supernatant liquor in tank W-10 are not favorable to the ferrous hydroxide carrying method, the concentration of salts being too high. A direct CuS precipitation on the acidified liquor may be used for treating this tank waste.

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

One Hanford-irradiated CaO slug was processed, being prepared for shipment as calcium chloride solution. Analyses have not been completed.

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

No runs were made.

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

No iron was processed.

10. Miscellaneous Work

Four Hanford-irradiated gold samples enclosed in aluminum were opened and the gold shipped. A curious phenomenon was noted: the bombardment had apparently caused the inside of the Al can to become coated with a thin film of gold. A portion of this was removed and dissolved for a study of its radiation. It was noted that the radiation is apparently long-lived. It has not been identified. A portion of the gold-plated aluminum was turned over to the Chemistry Division for further study.

Some C^{14} was extracted from irradiated $Pb(NO_3)_2$ samples submitted by the Physics Division. A report is being written on the results.

Irradiated TiO_2 was dissolved in sulfuric acid for a special shipment.

Waste samples of Element 61 from the Chemistry Division are being processed to remove the Y contamination, which we desire; the 61 will be returned to the Chemistry Division.

II. Tank Farm and Burial Ground:

1. Special Wastes

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

a. Twenty pots and fourteen drums were received from Chicago. The pots contained liquids contaminated with fission products. Seven drums contained 153.6 kg of uranium and the remainder hexone and aqueous solutions.

b. Two shipments of contaminated trash from K-25.

c. Five shipments of alpha-contaminated waste from Dayton.

d. The Ruthenium Process, this month, consumed 27,600 gallons of precipitated metal supernatant which contained an average of .005% uranium. The last ruthenium run, using supernatant from W-7 tank, has been completed. The average uranium content in the waste discharged was .0083%. A total of 104,400 gallons was used.

e. The Hot Pilot Plant transferred 47,706 kg of uranium and 8.37 grams of plutonium to tank W-4.

f. Samples of mud were taken from the bottom of the Settling Basin in an attempt to determine the amount of uranium precipitate. The average figure showed 1.05 mg of uranium per gram of wet mud. The total amount of uranium present in the Chemical Waste System, which includes tanks W-5 and W-6 and the Settling Basin, was estimated to be approximately 2,100 pounds.

2. Wastes Discharged to the White Oak Creek

The activity discharged to the White Oak Creek exceeded the five-curie-per-day limit on three occasions this month. This was due to the very poor conditions under which waste disposal was handled while the project to replace all the woodwork in the Settling Basin was carried out. Instead of discharging waste to the creek through the Settling Basin in the usual manner, it was diverted through the East Pond.

a. From Settling Basin

Gallons Discharged	To	Curies
9,122,000	White Oak Creek	33.25

b. From East Pond

9,907,000	White Oak Creek	55.47

c. The Retention Pond discharged 261,000 gallons of water carrying .08 curies of beta activity.

3. Waste Tank Inventory

CHEMICAL WASTE

Tank	Gallons Capacity	Gallons In	Gallons Out	Discharged To	Free Space
W-1-2-5-6	348,800	226,700	219,500	Settling Basin	227,200
		METAL WASTE			
W-4-7-8-9-10	713,000	7,000	27,600	Ruthenium Process	204,856
		Al-Pu WASTE			
W-3	41,300	888	0	---	38,184

4. Maintenance

a. The project to replace the above-ground transfer lines in the Tank Farm is about fifty percent complete. The line in the South Tank Farm is complete with the exception of tying in the steam to the jets, insulation, and painting of the supports.

b. Work is about seventy-five percent completed on the replacement of the woodwork in the Settling Basin. The inlet and exit weir boxes have been installed but not completely anchored. The baffles have not been replaced.

c. The Instrument Development Group has installed "V"-notched, stainless steel, weirs in the diversion box to the Settling Basin. Accuracy of these weirs is within two percent.

d. The first major overhaul of the 204 Area steam engines was done this month.

e. The W-5 concreted diversion box was found to be very badly eroded from acids having been sent through it. A project has been started to bypass this box. It is about thirty percent completed. The necessary ditches have been dug, but no connections have been made.

f. A new platform and shed were built at W-10 tank.

g. The new gauges have been installed on all the tanks in the Tank Farm and all have been calibrated except W-3. This will be done when there is enough liquid in the tank to bouy up the float.

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

RaLa run #26, scheduled to start on June 29, 1948, has again been postponed at the customer's request. The run is now tentatively scheduled to start on July 12, 1948. A small RaLa run is scheduled to start July 6, 1948, to provide a special source.

A pre-run check was made of Cells A and B. It was found that all thermocouples in the dissolver were corroded out of service and that one product carrier was out of line so that the cone adapter would not fit the elevator arms. These conditions are being corrected and the equipment should be ready for service by the first week in July, 1948.

The Hanford slug carrier was finished in June 22, 1948, and brought into 706-D for thermal conductivity tests. These tests were conducted by the Technical Division who were aided by 706-D personnel through the evening shifts. The preliminary results indicate the carrier to be satisfactory.

A scrubber installation is being made on the east side of Cell A to enable members of the Chemistry Division to scrub the dissolver off-gases for short-lived fission products. This installation should be completed by July 12, 1948, for use in conjunction with the next RaLa run.

C. ISOTOPE CONTROL DEPARTMENT

I. General:

The following table shows the number of shipments for May and June, 1948, and the total-to-date figure for all shipments made since August, 1946, the start of the Isotope Distribution Program:

	MAY 1948	JUNE 1948	TOTAL AUGUST, 1946, to JUNE, 1948, Inc.
Separated Material 706-D Area	222	268	2791
Unseparated Material 100 Area	59	59	1026
	281	327	3817

The 327 isotope shipments this month are greater than any month to date. In April, 1948, which had the most shipments of any previous month, there were 299 shipments.

II. Iodine Distribution:

Approximately 5,100 millicuries of radioiodine were shipped this month. This is an increase of about 500 millicuries over May, 1948.

III. Radio-cobalt:

Several new inquiries were received recently regarding very large orders for radiocobalt. In view of the large demand, irradiation at Hanford will be started as soon as definite information on the orders is received. Producing enough Co^{60} to fill all orders at one time will be considerably cheaper than producing each order singly.

Dr. William G. Myers of the Ohio State University has suggested that there would be considerable demand for radiocobalt prepared in such a manner that it might be used in standard radium needle sheaths.

Accordingly, an irradiation of cobalt needles will be made at Hanford to give sources comparable to radium sources now used in radium therapy.

IV. Sulfur 35:

Orders for sulfur 35 in the form of sulfide are still being held until a method of preventing oxidization of the sulfide to sulfate has been developed. Some customers have, however, been able to take S^{35} in the form of sulfate and where necessary perform the reduction to sulfide at the time the material is needed.

V. Fission Products:

Most of the long-outstanding orders for fission products either have been shipped or are scheduled to be shipped soon.

VI. Shipping Regulations:

The regulations for shipment of isotopes by motor freight have been accepted by the Interstate Commerce Commission so that it is possible to ship on some motor freight lines. The first motor freight shipment was made on June 30, 1948, to the Ford Motor Company.

Lately there has been considerable confusion in certain foreign shipments because the proposed air shipping regulations require that beta emitting isotopes be packaged with less than 10 mr/24 hours at the surface. One airline has recently declined to accept shipments packed under the current regulations of 15 mr/hr at the surface of the package. Since the proposed air shipping regulations require less than 10 mr/24 hours, all shipments of beta emitters would require much heavier containers than are now being used. Arrangements have been made with the Sub-committee on shipment of radioisotopes of the

National Research Council to revise the proposed shipping regulations. At the suggestion of the Committee, a working arrangement has been made whereby all strong beta emitters will be considered to be gamma emitters for the purpose of the shipping regulations, since only secondary radiation comes through the shipping shield.

VII. Oak Ridge Institute of Nuclear Studies:

Considerable demand from the radioisotope training school of the Oak Ridge Institute of Nuclear Studies has developed as classes began at the end of June, 1948. The use of radioisotopes should be greatly increased as the trainees go back to their regular places of employment.

VIII. Source and Fissionable Material Accountability:

As of June 1, 1948, the Source and Fissionable Material Accountability Office assumed the responsibility for all materials in the vault, Building 103. Shipments from other Atomic Energy Commission installations are now actually received by the Accountability Office except in those instances where material is "Hot" and must be delivered to the 105 Building Canal for storage. Whenever practical, shipments are sampled for analysis to verify the S. F. Material content.

In order to place responsibility for material lost or unaccounted for in Machine Shop operations, a new account has been set up for the "Hot Shop" in Building 101. The "Hot Shop" will comply with the S.F. Material Accountability Procedure in the future.

Following is a summary of Shipments and Receipts for the month of June, 1948:

RECEIPTS

Received From	Material	S.F. Content
Argonne National Laboratory	U.N.H. Solution	18.49 kg.
" " "	" "	10.85 kg.
" " "	" "	130.70 kg.
General Electric Co., Hanford	Slugs, Uranium	42.40 kg.
" " " "	" "	133.36 kg.
" " " "	Rods, "	291.66 kg.
" " " "	Thorium	250.27 kg.
Carbide & Carbon Chemicals, K-25	Uranium Waste	.04 kg.
Battelle Memorial Institute	Depleted Uranium	4.87 gr.
" " "	Enriched Uranium	4.73 gr.
U.S. Govt. Whse., Middlesex, N.J.	Thorium Nitrate	1,810.00 kg. Compound
Lindsay Light and Chem. Co.	Thorium Nitrate	45.36 kg. "
U.S.A.E.C., St. Louis	U_3O_8 Normal	28.85 kg. Uranium

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SHIPMENTS

Shipped To

Argonne National Laboratory	Slugs, Uranium	20.98 kg.
General Electric Co., Hanford	E. U. Alloy	683.78 gr.
" " " "	" "	685.96 gr.
" " " "	" "	673.49 gr.
" " " "	Normal U. Alloy	.03 kg.
Brookhaven National Laboratory	U_3O_8 Normal Uranium	.02 kg.

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