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AEC RESEARCH AND DEVELOPMENT REPORT

OPERATIONS DIVISION REPORT

FOR

MONTH ENDING JULY 31, 1948

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ORNL-119
Progress Report

OAK RIDGE NATIONAL LABORATORY

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OPERATIONS DIVISION REPORT

for

Month Ending July 31, 1948

PILE DEPARTMENT
CHEMICAL SEPARATIONS DEPARTMENT
RADIOISOTOPE DEVELOPMENT DEPARTMENT
ISOTOPE CONTROL DEPARTMENT

by

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J. A. Cox
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Date Received Series A: 8/9/48

Date Issued: AUG 13 1948

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ORNL-119
Progress Report

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ORNL-119
Progress Report

SUMMARY

1. Six ruptured slugs were located and discharged from the pile during the past month. These failures bring the known number of such incidents to forty-eight since the start of operations in November, 1943.

2. Isotope production continued normally throughout the month with only minor difficulties.

3. Further development work on the glacial acetic acid method for P^{32} production has been dropped because of poor yields. A comprehensive survey of possible methods is being made to determine which process to inventigate next.

4. Sulfur (S^{35}) isolated as the sulfide has been stabilized and is again being supplied to customers.

5. Two runs (Nos. 25 and 26) of RaLa were completed during the month. Run #25 contained 1,576 curies and was isolated in three parts of 1,409, 120, and 26 curies, respectively. These three gamma sources were used in the ARUU Program. Run #26 contained 2,419 curies and was shipped to Los Alamos on July 23, 1948. Run #27 is scheduled to start August 24, 1948.

6. There were 315 isotope shipments made in July as compared to 327 shipments during June. This brings the total since the start of the Isotope Distribution Program in August, 1946, to 4,132.

7. Design information for the new Isotope Production Area have been furnished to Patchen and Zimmerman. It is anticipated that construction will start in October, 1948.

[redacted]

A. PILE DEPARTMENT

I. Operating Data:

	July 1948	June 1948	Year-to-Date
Total Accumulated KWH	2,542,636	2,392,737	17,536,616
Average KW /operating hour	3790.17	3670.92	3756.92
Average KW/24-hour day	3417.52	3323.25	3431.09
Percent Lost Time	9.8%	9.5%	8.7%
Approx. Excess Pile Reactivity	170-175 inhours	190-200 inhours	
Slugs Charged	1989	2813	7359
Slugs Discharged	1852	64	4677
Product Made (grams)	92.80	87.33	640.03
Product Discharged (grams)	75.75	0.83	137.51

II. Pile Operation:

Six slugs with ruptured jackets were detected and discharged during the month.

Date	Channel No.	Days Exposure	Temp. Range	Slugs in Row
July 20, 1948	1077	1362	Approx. 125°C.	54
July 27, 1948	961	1400	Approx. 160°C.	54
July 28, 1948	1668	144	Approx. 210°C.	39
July 29, 1948	1459	1420	Approx. 175°C.	54
July 30, 1948	2874	1378	Approx. 125°C,	54
July 30, 1948	2879	1532	Approx. 100°C.	54

The ruptured slugs detected on July 20th and 27th were found during routine visual inspections of the metal channels in the pile. The other four ruptured slugs were indicated by high readings on the probe located in the exit air stream. The pile was scanned but the scanning operations did not definitely locate the position of the rupture because of the high background. However, they were located with little difficulty by visually inspecting all channels that gave readings slightly higher than background.

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With the exception of the slug in channel 2874 which had just ruptured at the time of its detection, the slugs were in an advanced state of oxidation varying from about one-fourth to almost complete oxidation.

Since the removal of the thirteen slugs that had ruptured during December, 1947, the detection of ruptured slugs by high exit air contamination has been difficult. Prior to December, 1947, the probe in the exit air stream read one to four mr per hour. It now reads twenty to thirty mr per hour even when there is no ruptured slug in the pile. Since the probe is currently the best method of detection, the slugs that have been detected recently have been in a more advanced state of oxidation.

The metal channels are to be visually inspected at least once per month even though this method is not likely to detect the early stages of a rupture. In addition to this, an attempt will be made to develop a better system of slug rupture detection.

The reason for these ruptures is not known. However, it is perhaps worthy to note that the average age of all ruptures slugs at date of rupture has been 534 days. This indicates that the age of a slug is not too accurate a measure of its probability of rupture since approximately sixty-five percent of the metal has been in the pile over 1,300 days and approximately eighty-five percent of the metal has been in the pile for over 1,000 days. These figures were slightly higher prior to the charging of 3.6 tons of additional metal on June 22, 1948.

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.

Nine hundred and sixty-eight X-slugs were discharged on July 13, 14, and 16, 1948, for 706-D Building.

The pile-down time is higher than usual this month due to the difficulties with ruptured slugs.

III. Fan Operation:

The fans have operated without any difficulty during the month.

IV. Radioisotopes:

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

	JUNE, 1948		JULY, 1948	
	Research	Radioisotopes	Research	Radioisotopes
Stringers 13, 14, and 16	53	126	13	92
Hole 22 (Pneumatic Tube)	27	3	24	7
All Other Holes	15	10	13	10
TOTAL BY GROUPS	95	139	50	109
TOTAL FOR MONTH	234		159	

At the end of July, 1948, there were 342 cans of target material in Stringers 13, 14, and 16 compared to 368 cans of material in these stringers at the end of June, 1948.

V. Experimental Work:

Another core was taken through the rear wall of the pile shielding this month. This was the third core sample taken for determination of

the effect of several years' bombardment by neutrons on the structural characteristics of the concrete.

A large number of special samples were prepared and irradiated for the ARUU Program. Five hundred and twenty slugs were discharged for the same program on July 6 and 7, 1948, and sent to 706-D Building for processing.

B. CHEMICAL SEPARATIONS AND ISOTOPE DEVELOPMENT DEPARTMENTS

I. Radioisotopes:

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

Thirty-six, seventy-two-gram, cans of irradiated tellurium were processed and approximately 3,900 millicuries of I^{131} were shipped. All product was within specifications.

Fission Product Iodine Development

Five dissolvings were made during the month for iodine development work. The alkaline coating removal (5% NaOH-10% $NaNO_3$) procedure was used in all runs. Several runs were needed to completely remove the last traces of mercury from the dissolver. The release of iodine from the dissolver improved considerably, but remained inconsistent. From eighty-eight percent to twenty-nine percent of the iodine was removed from the dissolver during

these runs. Primary distillation yields were approximately forty to fifty percent.

Various methods were used for purification of crude distillates in Room 10. In all cases, the crude distillates were made alkaline and the volume reduced by evaporation. In one case, nitrates were removed by reduction to ammonia in alkaline solution and iodine was removed by distillation from a twenty percent sulfuric acid solution with ferric sulfate oxidizing agent. Another procedure was also used in which iodine was oxidized to iodate with alkaline permanganate, nitrates distilled out, the permanganate reduced with phosphorus acid, and the iodine distilled. Both methods yielded good quality distillates with concentrations ranging from 1 mc/ml to 4 mc/ml.

Arrangements have been made with the Biology Division to have animal tests made on the products from the tellurium process and the new fission iodine process. These tests will be concerned primarily with chemical toxicity. Arrangements have also been made to ship fission iodine to various highly-qualified users, free of charge, for their appraisal.

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

Ten, 2000-gram, cans of irradiated sulfur were processed and approximately 3,800 millicuries of P^{32} were shipped.

The extraction equipment was shut down for one week and decontaminated for maintenance. The work accomplished included the repair of the leaking steam jacket on a transfer line, three bad steam traps, leaking steam and water valves, and torn insulation.

These repairs appeared to have eliminated to some extent the plugging of the transfer lines. Of the three runs made after the maintenance work was completed, only the first one showed signs of plugging.

Phosphorus Development Work

Yields on the acetic acid process run consistently twenty percent or less. No losses due to volatile phosphorus compounds were observed. It appears that the removal of P from S with glacial acetic acid is purely an extraction process with a very low distribution coefficient. With our large batches of sulfur a prohibitively large quantity of acetic would be required. No further work on this process is planned at this time.

A new sodium hydroxide extraction process is being tried. Also, a procedure for complete dissolution of sulfur in sodium hydroxide is also being investigated. A comprehensive survey of possible methods is being made to determine which process to investigate first.

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

No runs were made.

Beryllium Nitride Development Work

No further development work was done this month. Electronic equipment needed to complete the installation has not been received.

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

About 650 millicuries of carrier-free S^{35} as sulfate were produced during the month.

Approximately 800 millicuries of S^{35} as sulfide was produced during the month. A new procedure was used to provide sulfide in a solution which will resist oxidation to sulfate or thiosulfate. H_2S^{35} was scrubbed out in an oxygen-free barium hydroxide solution and stored under a nitrogen atmosphere. Tests have indicated that this method will allow less than ten percent oxidation of sulfide per month of storage. The amount of carrier has also been increased to further promote sulfide stability.

Analysis of this product was as follows:

Total Solids	-	10.3 mg/ml
Alkalinity	-	0.15 normal
Sulfate	-	0.11 mf/ml
Sulfide	-	0.61 mg/ml
Barium	-	8.50 mg/ml
Specific Activity	-	13.1 mc S^{35}/mg S.

Sulfate in this solution will be precipitated as it is formed. Instructions will be sent with each shipment giving recommendations for use of the material.

5. Fission Products

The installation of equipment inside the fission product column cell is substantially complete. Work is now being done on exterior connections to the panel board and installation of service lines. A cart for remote control withdrawal of fission product solutions from the cell has been designed and fabrication has started.

About 250 millicuries of Zr-Cb was produced during the month and shipped. The material shipped to non-project customers contained less than 150 alpha counts per mc of Zr-Cb; that sent to project users contained less than 200 alpha counts per mc Zr-Cb.

The purification of this Zr-Cb was accomplished entirely by a column process. As soon as the fission product cell starts producing material, Zr-Cb crude solutions of higher ratios of Zr-Cb/Pu will be produced, thereby simplifying the problem of purification. A column process is also being investigated for separation of Zr and Cb.

Approximately 400 millicuries of Ce^{144} was produced. The alpha content was zero, within the range of experimental error in alpha counting.

About 250 millicuries of Cs^{137} was produced, free from alpha contamination. Of this amount, 193 millicuries were shipped.

Approximately 100 millicuries of Y^{91} are in process. This material is being removed from "61" process waste which originally contained gross amounts of Pu.

Improvements to the working area on the third level, 706-D, have been completed. This work includes a lead-lined hood and a lead-covered table.

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

Tank W-7 supernatant liquor has been completely processed. Two experimental runs on the volume reduction procedure, carrying Ru on precipitated sulfur, have been completed. The yield is approximately ninety-five percent for this step.

The overall yield on ferrous hydroxide carrying of Ru from alkaline waste liquor has been disappointing and inconsistent, ranging from ten percent to fifty percent. Direct carrying of Ru

from acidified W-10 waste liquor with CuS will be tried when this tank is processed.

Approximately two curies of Ru^{106} are in process of volume reduction. In addition to this material, 800 millicuries of Ru^{106} from Hanford slugs ("43" process waste) are on hand.

The Ru purification hood has been completed and a new fan installed to provide adequate ventilation. The glassware will be installed and brick barricade constructed during the coming month.

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

Analysis of the Hanford-irradiated preparation reported last month is as follows:

Total Volume	-	400 ml
Concentration	-	1.14 mc/ml
Calcium	-	55.1 mg/ml
Total Solids	-	169.4 mg/ml
Specific Activity	-	27 mc Ca^{45}/gram Ca
Spectrographis Analysis	-	No important contaminants.

Six, 0.3 gram, batches of scandium were purified and put in the pile for production of carrier-free Ca^{45}.

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

No runs were made.

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

One, twelve-gram, batch of Hanford-irradiated iron was processed to produce ferric chloride solution. Analysis is as follows:

Total Activity (gross beta, Fe^{59})	-	113 mc
Iron	-	54.8 mg/ml
Concentration	-	0.49 mc Fe^{59}/ml
Specific Activity	-	8.9 mc Fe^{59}/gram Fe.

Analyses for Fe^{55} have not been completed by the Chemistry Division.

10. Miscellaneous Work

Three tubes of NaCl and three of HgO were prepared and sent to Hanford for irradiation.

One unit of nickel irradiated in the ORNL pile is in process of purification. A very large amount of Co^{60} impurity was formed, obscuring the Ni^{59-63} activity. The by-product Co^{60} will also be available. The Ni^{59-63} was counted on the beta proportional counter developed by C. Borkowski; this is the only counter which gives reasonable figures for the assay.

One unit of $Hg(NO_3)_2$ is in process of purification. This material is contaminated with aluminum which occurred as a result of corrosion of the isotope can during irradiation. The mercury will be separated as the sulfide and converted to the nitrate for shipment.

II. Tank Farm and Burial Ground:

1. Special Wastes

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

a. Twenty-two pots and twenty-four drums were received from Chicago. The pots contained solutions with fission products only. Five drums contained 182.88 kg of uranium. The remainder contained chemical wastes.

b. One shipment of contaminated trash was received from K-25.

c. K-25 transferred 800 gallons of liquid waste to the Tank Farm.

d. Four shipments were received from Dayton for burial.

e. The Hot Pilot Plant transferred 37.18 kg of uranium to W-4.

f. The Semi-Works transferred 20.62 kg of uranium to W-4.

g. 706-D transferred 9,025 gallons of UNH to W-9.

h. Two shipments of contaminated trash from the Oak Ridge Institute of Nuclear Studies were buried at the West Burial Ground.

i. Three, five-gallon, stainless steel drums from Chicago, containing radium waste (10.1 mg Ra) were buried 250' west by 0-10' south. A special marker has been erected on this spot.

2. Wastes Discharged to the White Oak Creek

The East Pond was used during the first eight days this month while the Settling Basin woodwork replacement project was being carried out.

On July 8, 1948, water was again turned into the Basin. About twenty-nine hours later it began to overflow to the Creek. The delay was due to the level of the Basin having been previously pumped down.

The new calibrated "V" notched weirs in the Diversion Box ahead of the Basin were put into service this month. The present flow of water, as shown by the new weirs, is about 432,000 gallons per day, indicating that the previous Diversion Box calibration was approximately twenty-four percent too high and that the reported Settling Basin activity discharge was correspondingly high.

a. From Settling Basin

Gallons Discharged	To	Curies
9,632,000	White Oak Creek	28.89

b. From East Pond

3,759,000	White Oak Creek	7.68

c. The Retention Pond discharged 383,000 gallons of water carrying .07 curies of beta activity.

3. Waste Tank Inventory

Tanks	Gallons Capacity	Gallons In	Gallons Out	Discharged To	Free Space
		CHEMICAL WASTE			
W-1-2-5 -6	348,800	101,700	125,700	White Oak Creek	164,800
		METAL WASTE			
W-4-7-8 -9-10	713,000	1,792	0	-	178,800
		AL-PU WASTE			
W-3	41,300	0	0	-	38,776

4. Maintenance

a. The project to replace the transfer lines at the Tank Farm is about ninety-five percent complete. Some painting and insulation work remains to be done.

b. The project to bypass the W-5 Diversion Box is complete. All the lines which were broken during excavation have been repaired.

c. All the work on the wood replacement project at the Settling Basin has been completed except the replacement

of the baffles. This will be done when the riggers become available.

d. The new caustic shed at W-10 has been painted and the steam coils installed.

e. A CL-1A jet was installed at W-5 platform, replacing a CL-1.

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

The equipment repairs mentioned in last month's report were completed prior to the start of run #25.

To provide special sources for the ARUU Program run #25 was started on July 6, 1948, and turned over to the Atomic Energy Commission on July 17, 1948, after decay equilibrium had been reached. The run was packaged into three separate carriers with the product contained in especially adapted cones, plugs, and plug shields. No difficulty was encountered in this run other than a slight delay when a sparger line failed in the product evaporation tank. This condition was remedied in a short time and L.S.T. was reached on July 12, 1948, at 0745. The three product carriers contained approximately 1,409, 120, and 26 curies, respectively.

The analytical summary of this run is tabulated below:

	CURIES	PERCENT
Total Curies Dissolved	2,328	100
Cell A Losses	738	31.68
Cell B Losses	78	3.35
Total Accounted Losses	816	35.03
Approximate Quantity Shipped (Total of 3 products)	1,576	67.7
Material Balance		95.58
Losses Unaccounted For		4.42

Run #26, for Los Alamos, was begun on July 13, 1948, immediately following the completion of run #25. All the salvageable wastes and remains of urnaium slugs from run #25 were processed in this run.

L.S.T. was reached on July 2, 1948, at 1520 and the product was shipped on July 23, 1948. The product contained 2,419 curies or 3,387 Los Alamos units as measured by "Skyshine" direct radiation measurements. No difficulties were encountered other than minor instrument failures which were remedied on an emergency basis.

The analytical summary of run #26 follows:

	CURIES	PERCENT
Total Curies Dissolved (Includes wastes from run #25)	4,501	100
Cell A Losses	1,464	32.52
Cell B Losses	184	4.09
Total Accounted Losses	1,648	36.61
Approximate Quantity Shipped	2,419	53.74
Material Balance		90.58
Losses Unaccounted For		9.42.

The HCl-ether wastes of both runs were packaged and stored for use in the processing of strontium. The tanks in both cells have been washed and the hot lines and sample blisters replaced or decontaminated for the next run which is scheduled to start on August 24, 1948.

The Chemistry Division used the new scrubber installation to scrub ten percent of the dissolver gases from three slug dissolvings during runs #25 and #26. The equipment operated satisfactorily and gave a large yield of short-lived fission products.

The thermal conductivity tests on the Hanford slug container show it to be satisfactory for the transportation of "W" slugs from Hanford to Oak Ridge. As soon as the Atomic Energy Commission has approved

the overall program, Hanford-irradiated material will be used for the production of RaLa

C. ISOTOPE CONTROL DEPARTMENT

I. General:

During the month of July there were 315 radioisotope shipments, a decrease of twelve shipments from the month of June when there were 327. This is the second largest number of isotope shipments we have had in any month to date.

The decrease is probably due to the vacation season and to the fact that most of our shipments are made on Tuesday and Wednesday. There were effectively five weeks in June compared with four weeks in July.

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

	JUNE 1948	JULY 1948	TOTAL August, 1946, to July, 1948, Inc.
Separated Material 706-D Area	268	237	3,028
Unseparated Material 100 Area	59	78	1,104
	327	315	4,132

II. Radio-Cobalt:

In addition to the cobalt-nickel alloy wire which will be irradiated to give sources for use in standard radiological equipment now used in hospitals, several 5/16-inch diameter cylinders of fairly pure cobalt have been obtained from a commerical concern. These cylinders have been prepared by powder metallurgy from pure cobalt powder. It appears that such cylinders will be useful in preparing large sources of cobalt 60 in the order of 10 to 1,000 curies.

III. Phosphorus 32:

The demand for radioactive phosphorus has varied considerably lately. In the last week of July there was more P^{32} (1,466 mc) shipped than in any previous week.

IV. Shipping Regulations:

Some trouble has been encountered with Pan American Airways in the shipment of radioisotopes to South America. This airline refuses to recognize the present regulations used for air shipment of radioisotopes and negotiations are now under way to have the regulations accepted. Shipment of radioisotopes to Argentina is held up because of this.

V. Fission Products:

Eu^{155} has become occasionally available as a by-product of research and the first shipment of this material was made to Dr. J. G. Hamilton of the University of California.

VI. Isotope Production Area:

The new Isotope Production Area is being designed by Patchen and Zimmerman. Design information has been furnished them by the Operations and Engineering, Maintenance, and Construction Divisions. A plot plan has been prepared and submitted to the Atomic Energy Commission for approval. It is anticipated that actual construction will start in October, 1948.

This area will be located to the east and north of the present 706-D Building. A 250-foot off-gas stack will be provided to handle the radioactive wastes from the 706-C and 706-D operations, as well

as the new facilities. In addition to the stack, this new area will contain the following structures:

1. Fans and fan enclosures near base of stack.
2. A one-story (about 8,000 sq. ft.) building to house analytical, storage, packing, shipping, and decontamination facilities.
3. A two-story (about 7,000 sq. ft.) building housing locker rooms, offices, etc.
4. Four one-story (about 800 sq. ft. each) buildings to house processing equipment.
5. One thirty-foot high structure of about 900 sq. ft. floor space adapted to column extraction of radioisotopes.

The plot plan allows for adequate expansion of all facilities at some later date.

VII. Source and Fissionable Material Accountability:

The Inventory and Material Balance Report for the month ending June 30, 1948, was submitted to the Commission under date of July 16, 1948.

The S.F. Material Accountability Procedure is in process of being revised by the Methods and Procedures Section and it is contemplated the revised procedure will be completed and distributed in the near future.

Following is a summary of Shipments and Receipts of S.F. Materials for the month of July, 1948:

RECEIPTS

Received From	Material	
Argonne National Laboratory	U.N.H. Solution	46.50 kg.
" " "	U.N.H. Solution	3.94 kg.
" " "	U.N.H. Solution	37.07 kg.
" " "	U.N.H. Solution	24.06 kg.
" " "	X Slugs	3.50 kg.
A.E.C., O.R.L.D.	Normal Uranium	1.05 kg.
Carbide & Carbon Chemicals Co., K-25	Waste Scrap	.02 kg.
General Electric Co., Hanford	W Size Slugs	42.40 kg.
Battelle Memorial Institute	Thorium Metal	61.30 kg.
General Electric Co., Hanford	Plutonium in W Slugs	8.37 gr.
A.E.C., O.R.L.D.	Enriched Urnaium	1,057.00 gr.
" "	Enriched Uranium	1,059.00 gr.
Carbide & Carbon Chemicals Co., Y-12	Enriched Uranium	64.45 gr.

[redacted]

SHIPMENTS

Shipped To		
Argonne National Laboratory	X Slugs	20.98 kg.
Carbide & Carbon Chemicals Co., Y-12	Enriched Uranium	220.13 kg.
General Electric, Hanford	Enriched Uranium	552.25 gr.
General Electric, Hanford	Enriched Uranium	2.60 gr.

[redacted]