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Progress Report

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OAK RIDGE NATIONAL LABORATORY

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OPERATING DIVISION REPORT

for

Month Ending February 29, 1948

PILE OPERATIONS  
SEPARATIONS  
RADIOISOTOPES

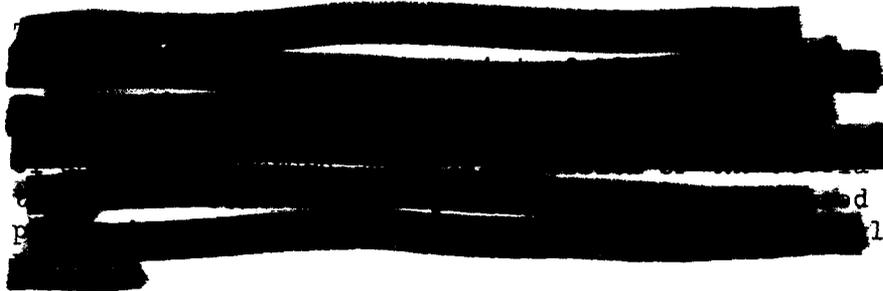
by

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

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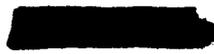
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3.

SUMMARY

1. Pile operation was normal throughout the month. No ruptured slugs were detected.
2. The excess reactivity remains about seventy inhours.
3. The  $\text{Be}_3\text{N}_2$  canning program has been completed. Work has started on canning  $\text{Ca}(\text{NO}_3)_2$  for exposure in the Clinton Pile to produce  $\text{Cl}^{14}$ .
4. RaLa run was started on February 24, 1948. Shipment is scheduled for March 5, 1948. The results will be reported next month.
5. The modifications to the pressure process equipment for  $\text{P}^{32}$  have been completed. The initial runs indicate a great improvement in operation.
6. Two hundred and three isotope shipments were made during the past month to bring the total to 2,641 since the start of the Isotope Distribution Program in August, 1946.

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A. 100 AREA OPERATION

I. Operating Data:

	<u>February</u> <u>1948</u>	<u>January</u> <u>1948</u>	<u>Year to Date</u>
Total Accumulated KWH-----	2,437,030	2,365,869	4,802,899
Average KWH/operating hour-----	3790.63	3760.12	3775.18
Average KWH/24-hour day-----	3501.43	3179.93	3335.35
Percent lost time-----	7.6%	15.4%	11.6%
Approx. Excess Pile Reactivity---70-75 inhours---70-75 inhours-----			
Slugs Charged-----	997	1033	2030
Slugs Discharged-----	997	1099	2096
Product Made (grams)-----	88.94	86.35	175.29
Product Discharged (grams)-----	22.46	26.62	49.08

II. Pile Operations:

The pile operation was normal throughout the month with the usual scheduled shutdowns for the removal and insertion of samples, discharge of slugs, and other miscellaneous work. The testing of shielding materials at the West Core Hole increased slightly this month and accounts for about two percent of the total lost operating time.

No trouble was encountered with ruptured slugs. The visual survey of all loaded channels was completed. All wooden ends of the front face shielding plugs have been renewed with the exception of those channels containing thermocouples.

The Technical Division assisted by the Instrument Department are attempting to develop a sensitive air velocity measuring element which will detect a slight change in the cooling air velocity caused by the swelling of slugs. If this instrument can be made sufficiently sensitive, a swollen slug will be detected before it ruptures. More information on this problem will be available in a month or so.

The excess pile reactivity remained unchanged during the month. At the present time there are about seventy inhours available.

III. Fan Operation:

The ductwork on the discharge side of the No. 1 fan (steam-driven stand-by fan) which had been leaking and causing air contamination in the cell has been repaired.

The No. 2 fan, which was highly contaminated with radioactive material due to recent slug ruptures, was partially decontaminated this month so that adequate time can be spent in the cell to periodically check the condition of the fan bearings.

An oil line to the motor bearing of the No. 2 fan failed three times this month due to vibration and strain. There have been no further failures since the strains in this line were properly relieved.

The No. 3 fan installation operated satisfactorily throughout the month.

IV. Radioisotopes:

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

	<u>JANUARY</u>		<u>FEBRUARY</u>	
	<u>Research</u>	<u>Radioisotopes</u>	<u>Research</u>	<u>Radioisotopes</u>
Stringers 13 and 14	12	80	10	95
Hole 22 (Pneumatic Tube)	49	0	65	0
All other holes	9	20	7	25
TOTAL BY GROUPS	70	100	82	120
TOTAL FOR MONTH	170		202	

As of February 29, 1948, there were 351 cans of target material in Stringers 13, 14, and 16, compared to 321 cans of target material at the end of January, 1948.

V. Miscellaneous:

1. Beryllium Nitride Canning

The beryllium nitride canning program was completed during February. The final 624 cans have been tested and packed and are awaiting A.E.C. approval for shipment to Hanford Engineer Works for C<sup>14</sup> production. Since the reworking of reject material proved to be rather difficult, the program was considered to be completed when the rejects had been reduced to a minimum. The final results of the laboratory canning program are listed below:

Number of Be <sub>3</sub> N <sub>2</sub> Cans Shipped to Hanford	550
Number of Be <sub>3</sub> N <sub>2</sub> Cans to be Shipped to Hanford	<u>624</u>
TOTAL Be <sub>3</sub> N <sub>2</sub> Cans Passing Inspection	1174
Number of Be <sub>3</sub> N <sub>2</sub> Cans Rejected	19

2. Calcium Nitrate Canning

The calcium nitrate canning program was reactivated during the month. The program is to can 1,950 pounds of calcium nitrate which will produce approximately 6,900 slugs for C<sup>14</sup> production in the Clinton Pile. Approximately 2,000 pellets (two pellets used per can) have been pressed and one hundred cans have been welded but have not been tested.

## B. 706-D AREA

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

No shipments were made this month. Run #24 was started on February 24, 1948, and is still in progress. No difficulties have been encountered to date. The shipment is scheduled to be made on March 5, 1948.

The preliminary design studies of a container for transporting Hanford-type slugs to eliminate the present use of "X" slugs, which was reported last month, will be completed during the coming month. The main problem is to provide a simple cooling system for the slugs while they are in transit.

A dummy run using twelve "cold" Hanford slugs was made. This run indicated a necessity for minor alterations to the existing operating procedures. The changes include greater coating removal time and more thorough washing after coating removal to completely remove the silica bonding material used in the "W" slug canning procedure. Further tests are to be made following the completion of Run #24.

II. Radioisotopes:1. Iodine ( $I^{131}$  - 8d)

Twenty-eight, seventy-five-gram cans of irradiated tellurium were processed this month and approximately 2,650 millicuries of  $I^{131}$  were shipped. All of the product was within specifications.

Iodine Development Work

Two dissolvings were made in the tantalum-lined dissolver, one using three, sixty-nine-day slugs and the other, three,

eighty-nine-day slugs. The iodine yield from the dissolver to the scrubber and catch tank was sixty-five percent and the primary distillation yield was seventy percent. In the second run, sodium hydroxide solution alone was used in the distillate receiver instead of hydroxide sulfite mixture; no loss in efficiency was noted.

Although a total of about 3,600 millicuries of  $I^{131}$  was collected, it was discarded since purification facilities in Room 10 are not yet complete. Most of the glassware has been fabricated for the purification apparatus, the supporting framework installed for concentrating the primary iodine distillates from the cell.

The cell apparatus was successfully decontaminated to permit the repair of several leaks in cell piping and the replacement of a tantalum thermowell.

Three dissolvings of three slugs each were made for the Chemistry Division's solvent extraction equipment. These slugs were too old to contain any  $I^{131}$ .

UNH solution was supplied to the all-column fission product unit; especial emphasis was placed on eliminating traces of aluminum from the dissolver solution, since aluminum contamination may interfere with the rare earth separations.

Development work on the chemistry of the  $I^{131}$  final purification process has been directed toward the elimination of the small amounts of nitrates that occur in the primary (cell) distillates. It was found that traces of nitrates and nitrites

[REDACTED]

could be eliminated as ammonia by boiling the alkaline concentrate with ferrous sulfate. Following this treatment, the solution is acidified with sulfuric acid, excess ferric sulfate is added, and the iodine distilled off.

2. Phosphorus (P<sup>32</sup> - 14.3d)

Approximately 2,750 millicuries of P<sup>32</sup> were shipped this month. This product was obtained from eight, 2000-gram cans of irradiated sulfur. Six of these cans were processed in temporary equipment using the fuming nitric acid extraction method. The remaining two cans were extracted by the pressure process method in Cell 5, Building 205, after the equipment alterations had been completed.

The extensive alterations to the pressure process equipment which were described in last month's report were completed on February 20, 1948. This equipment, with one exception, is now operating satisfactorily. An improved method of loading the sulfur cans into the melter is needed to reduce the radiation hazard. This problem is being studied.

Phosphorus Development Work

No further development work was done on the acetic acid process during this period.

3. Carbon (C<sup>14</sup> - 5100y)

No material was processed this month. An adequate supply is on hand to meet current demands.

C<sup>14</sup> Development Work - (From B<sub>2</sub>N<sub>2</sub>)

Construction work on the revision of 204 Building Annex and installation of equipment has proceeded in a very satisfactory manner. The installation is about thirty percent completed.

4. Sulfur (S<sup>35</sup> - 87.1d)

No S<sup>35</sup> was separated during the month, there being an adequate supply on hand.

5. Fission Products

Three runs were completed in the all-column fission product plant, using UNH solution from the tantalum-lined dissolver. The slugs used in two of these runs were in the pile 900 days; slugs for the next run and a run now in process were ninety days in the pile. The equipment continued to operate satisfactorily and the search for optimum processing conditions occupied main attention during the month.

A five percent oxalic acid solution has been used to remove zirconium-columbium, but the eluate has considerable alpha contamination. It is planned to try 0.4% oxalic acid as an eluant, since this concentration has been reported to remove Zr-Cb without eluting Pu and Am. Recent work on the decontamination of the Ta-lined dissolver shows that some of the Zr-Cb is held up on the tantalum lining and, therefore, never reaches the columns. About forty percent of Zr-Cb is lost in waste effluents from column No. 1. Approximately 2.5 curies of crude Zr-Cb eluate is on hand and about 500 millicuries of this is in process of purification.

Yttrium has been removed from the all-column plant and purified by various methods involving extractions with TTA-benzene and separations on small auxilliary resin columns. An ample stock of crude yttrium is on hand and is being purified for shipment.

Some improvement has been made in handling the rare-earth fractions. The citrate R-E eluate from column No. 1 is now being re-absorbed on both columns, two and three, rather than column No. 3 alone, thereby, cutting losses in effluents from eighty percent to about thirty percent. The entire R-E (excluding cerium) fraction is then being eluted from columns Nos. 2 and 3 and an attempt is being made to separate the various components on auxilliary resin columns outside the cell. So far, separation of individual R-E components has not been very successful.

Little work has been done on the recovery of Ba, Sr, and Ce; however, considerable quantities of crude eluates are on hand for purification when time and space are available.

6. Ruthenium (Ru<sup>106</sup> - ly)

The operating group accepted responsibility for the Ru<sup>106</sup> operation on February 13, 1948. The group made eighteen ferrous sulphate concentrations on 8,100 gallons of waste from W-7 tank. The laboratory results indicate a yield of about 1,000 millieuries of ruthenium. The results are not too accurate, however, because of the necessity of sampling and analyzing a slurried precipitate rather than a true solution.

It was decided that the existing equipment should be altered in order to provide greater safety, from the standpoint of radiation, for the operating personnel. The processing was discontinued and the equipment was decontaminated and turned back to the Isotope Development Group for modifications.

Ruthenium Development Work

The Ru<sup>106</sup> operation in the Tank Farm Area was turned over to the regular operating group on February 13, 1948. A total of twenty-seven runs were made and the ferrous hydroxide slurry containing Ru<sup>106</sup> was stored in glass carboys and stainless steel drums to await distillation. The average recovery of Ru<sup>106</sup> from waste solution was thirty-eight percent and measurements indicate that an increase of eighteen percent could be obtained with longer settling periods; however, it has been decided to take this loss to cut down cycle time.

Distillation of Ru<sup>106</sup> from the iron slurry has been started. Recovery of ruthenium has been low and the distillate contaminated with unidentified material which distills over from the H<sub>2</sub>SO<sub>4</sub> - KmnO<sub>4</sub> solution.

7. Calcium (Ca<sup>45</sup> - 180d)

A batch of Hanford-irradiated calcium carbonate is being purified. This will be prepared finally as calcium nitrate; an adequate supply of Ca<sup>45</sup> as the chloride is on hand.

Revisions to the hot hoods in 706-D have been completed and some carrier-free Ca<sup>45</sup> will be separated from scandium, probably during March.

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8. Strontium (Sr<sup>89,90</sup> - 55d, 30y)

No material was processed this month. The HCl-ether waste from RaLa run No. 23, which is too radioactive to be processed at the present time, was stored for future use.

9. Iron (Fe<sup>55-59</sup> - 4y, 44d)

Four side hole tubes containing iron were shipped to Hanford for irradiation. One tube each of Fe<sup>54</sup> and Fe<sup>58</sup>, enriched isotopes from Y-12, were canned and sent to Hanford.

10. UX<sub>1</sub> (Th<sup>234</sup> - 24.5d)

A special sample of 111 millicuries of UX<sub>1</sub> separated from K-25 residue was sent to K-25.

11. Zinc (Zn<sup>65</sup> - 250d)

There was no further work on separation of Zn<sup>65</sup> from copper cyclotron targets during this period.

12. Miscellaneous Materials

A ten-gram sample of thallic nitrate was dissolved and prepared for distribution.

A 0.5-milligram sample was also dissolved and portions shipped to various requestors.

13. Organic Synthesis

Two shipments of labeled (C<sup>14</sup>) methanol have been made during the past month from material that was prepared by the Chemistry Division.

III. Tank Farm and Burial Ground:

1. Tank Farm

(a) The program to provide waste storage space for the Hot Pilot Plant, which was started last month, was continued

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but not completed as had been anticipated. Bad weather and mechanical difficulties encountered in pumping the waste slurry from W-3 to W-4 delayed the work. This work, however, is expected to be completed during the coming month.

In carrying out this program, 24,800 gallons of precipitated uranium equipment supernatant were decanted from W-3 and W-4 to the chemical waste system. One batch of 7,100 gallons contained .5 percent uranium and the other batch of 17,700 gallons contained .039 percent uranium.

- (b) To provide more metal waste storage, a uranium precipitation program has been completed in W-10. A total of 9,800 gallons of fifty percent NaOH was added to this tank and after a period of two to three months of settling, the supernatant will be removed, thus, leaving several thousand gallons of free space.
- (c) The ruthenium separation program consumed 16,250 gallons of supernatant from metal storage tank W-7. This solution which contained approximately .05 percent uranium was discharged into the chemical waste system.
- (d) Progress has been slow on the installation of the new W-12 tank due to bad weather. At the end of the month, work was resumed. It is about forty percent complete.
- (e) A total of nineteen, fifty-liter pots and eleven, fifty-five-gallon drums were received from Chicago this month. All of the pots contained an aqueous solution contaminated with fission products. The drums contained 86.93 kilograms uranium.

Some shipments of radioactive wastes arrive here with the exterior of the container and occasionally the truck bed grossly contaminated. An active campaign is at present being conducted to eliminate this needless hazard.

- (f) The Hot Pilot Plant, Building 205, transferred 106.08 kilograms of uranium as UNH to W-3 metal storage. The Isotope Development Department transferred 3.06 pounds of uranium to W-4. The 706-D operation jettied 43.54 kilograms of uranium from a test run of "W" slugs to W-9 plus 2,466 gallons of UNH solution from barium run No. 24.
- (g) A large number of poles supporting the various pipe lines in the Tank Farm were found to be rotted at the ground. A replacement program has been started.
- (h) The Health-Physics group has recommended that the procedure for calculating the curies of beta activity discharged from the Settling Basin be changed. Starting this month, all the calculations will be based on this recommendation. Effectively, this change lowers the allowable beta count by about seven percent.
- (i) The following is a listing of the movement of liquids in the Tank Farm for February, 1948:

<u>WATER WASTES</u>					
<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Feb.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-1 & 2	8,800 gal.	142,000 gal.	142,000 gal.	Settling Basin & Chemical Waste System	8,800 gal.

Approximately thirty percent of this 142,000 gallons was routed to the chemical waste system and the remainder went to the Settling Basin. It is estimated that about fifty percent of this water originated at Building 115. The remainder came from the other waste sewers at Buildings 105 and 205.

CHEMICAL WASTES

<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Feb.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-5 & 6	4,000 gal.	151,200 gal.	186,000 gal.	Settling Basin	167,400 gal.

Approximately twenty percent of the chemical waste was precipitated metal supernatant, fifty percent from 706-A, C, and D, and the remainder from the various warm sewers via tanks W-1 and W-2.

METAL WASTES

<u>Tanks</u>	<u>Capacity</u>	<u>Est. Amt. Rec'd-Feb.</u>	<u>Disposed Of</u>	<u>Discharged To</u>	<u>Free Space</u>
W-3-4-7 -8-9-10	755,700 gal.	4,000 gal.	17,700 gal. (precipitated U supernatant)	Settling Basin	177,760 gal.

Seventy-three percent of the metal waste received was from the RaLa operation. The remainder originated at the Hot Pilot Plant, Building 205.

SETTLING BASIN

<u>Total Est. Discharge</u>	<u>Total Curies Discharged</u>	<u>Beta counts/min/ml Average High Low</u>			<u>Discharged To</u>
28,249,000 gals.	72.96	203	953	5	White Oak Creek

RETENTION POND

526,000 gals.	.06	8	54	0	White Oak Creek
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2. Burial Ground

(a) Special Burials

- (1) Five alpha contaminated Dayton Shipments.
- (2) One alpha contaminated Chicago shipment.
- (3) A shipment of alpha contaminated material from Schenectady and Rochester.
- (4) The contents of six, sixty-gallon drums from Building 205.
- (5) One truckload of material, alpha contaminated, from A.E.C., Oak Ridge.

(b) Stored at Burial Ground

- (1) Several pieces of contaminated electrical equipment from the Semi-works, 706-A.
- (2) A truckload of contaminated piping from Building 205.

(c) Routine Burials

Three hundred and ninety-three red cans of contaminated trash from the Restricted Area.

C. RADIOISOTOPE PRODUCTION AND SHIPMENTS

I. General:

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

[REDACTED]

	JANUARY 1948	FEBRUARY 1948	TOTAL AUGUST, 1946, to FEBRUARY, 1948, Inc.
Separated Material 706-D Area	174	152	1869
Unseparated Material 100 Area	49	51	772
TOTAL	223	203	2641

Included in the February shipments are eight to institutions  
in foreign countries.