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OPERATIONS DIVISION MONTHLY REPORT

FOR

MONTH ENDING DECEMBER 31, 1952

AEC RESEARCH AND DEVELOPMENT REPORT

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OPERATIONS DIVISION MONTHLY REPORT

for

Month Ending December 31, 1952

by
M. E. Ramsey

DATE ISSUED

FEB 21 1953

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CONTENTS

	Page
SUMMARY	1
REACTOR OPERATIONS DEPARTMENT	3
Operating Data	3
Reactor Operations	3
ORNL graphite reactor	3
Summary of operations during 1952	3
Low-intensity test reactor	6
Summary of operations during 1952	6
Filter House	7
Summary of operations during 1952	8
Fan House	8
Summary of operations during 1952	8
Radioisotopes	8
Summary for 1952	8
Water-Demineralization Building	8
Summary of operations during 1952	9
CHEMICAL SEPARATIONS AND RADIOISOTOPE DEVELOPMENT DEPARTMENTS	10
Radioisotopes	10
Iodine (I^{131} – 8 d)	10
Summary of operations during 1952	10
Iodine development work	10
Summary of development during 1952	10
Phosphorus (P^{32} – 14.3 d)	11
Summary of operations during 1952	11
Summary of development during 1952	11
Carbon (C^{14} – 5740 y)	12
Summary of development during 1952	12
Fission products – summary of development during 1952	12
Tritium (H^3 – 12.4 y)	14
Tritium and helium – summary of development during 1952	14
Strontium purification	14
Sr^{90} source fabrication – summary of development during 1952	14
Precipitation process, Building 3515	15
Source assembly station, Building 3013	15
Gamma source loading – summary of development during 1952	15
Miscellaneous source fabrication – summary of development during 1952	15
Detection of Co^{58} in irradiated nickel	16

[redacted]

[redacted]

Europium target material – summary of development during 1952 16
Processed radioisotope production 16
Production summary for 1952 16
Special preparations 17
Summary of development during 1952 17
Hot chemistry cell – summary of development during 1952 17
Capryl alcohol as an extractant – summary of development during 1952 17
Separations from cyclotron targets – summary of development during 1952 17
High specific activity short-lived isotopes from the LITR – summary of development during 1952 18
Extraction of iron with TBP – summary of development during 1952 18
Underground storage of hot material – summary of development during 1952 18
Miscellaneous work 18
Summary of development during 1952 18
Radioactive-Waste Disposal 18
Waste discharged to White Oak Creek 19
Chemical-waste evaporator 20
Waste-tank inventory 20
Summary of radioactive-waste disposal during 1952 20
RaLa (Ba^{140} – 12.5 d) 21
Summary of operations during 1952 22

RADIOISOTOPE SALES DEPARTMENT 23

Hanford Irradiations 23
Cyclotron Radioisotopes 23
Activation Analyses 23
SF Material Control 25

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OPERATIONS DIVISION MONTHLY REPORT

SUMMARY

The activities of the Operations Division for the month ending December 31, 1952 are summarized and indexed below. In some instances, a comparison with 1951 and other previous yearly operational data is also given.

1. Lost ORNL graphite reactor operating time averaged 9.7% for December, compared with 10.4% for the year of 1952 and 10.0% for the year of 1951 (p. 3).

2. One ruptured slug was detected and discharged without difficulty. There were 13 slug ruptures in 1952, compared with 15 in 1951 (p. 3).

3. The difficulties encountered in the fabrication and the use of aluminum-silicon bonded slugs are reviewed (p. 4).

4. The LITR operated very satisfactorily throughout the year, with a downtime of 16.8% (p. 6).

5. Radioactive iodine shipments are averaging approximately 40 curies per month. Shipments for 1952 totaled 488 curies, compared with 467 curies in 1951 and 311 curies in 1950 (p. 10).

6. Radioactive phosphorus shipments totaled 256 curies in 1952, 264 curies in 1951, and 212 curies in 1950 (p. 11).

7. A total of 5.5 curies of C^{14} was produced during the year. The highest isotopic ratio was 28% (p. 12).

8. About 38 curies of Sr^{90} and 100 curies of Cs^{137} have been separated by the precipitation process designed for separating Cs^{137}, Sr^{90}, Ru^{106}, Ce^{144}, and the remainder of the rare earths (p. 12).

9. Almost all the equipment required for the kilocurie source-loading operation has been received (p. 14).

10. Satisfactory techniques for making thin $Zr\text{-}H^3$ targets have been developed (p. 14).

11. Preparation of satisfactory europium targets for production of high-intensity gamma sources is being studied (p. 16).

12. The beta-curie discharge to White Oak Creek was 498 curies for 1952, 172 curies for 1951, and 187 curies for 1950. The increase was largely due to spills. Methods of removal of the radioactivity from the process water stream are being evaluated (p. 21).

13. The disposal of radioactive wastes by storage in pits dug in the soil appears to offer a solution to the radioactive waste storage problems (p. 21).

14. The present RaLa equipment is not reliable for future RaLa production, and the need for permanent RaLa facilities is discussed (p. 22).

15. There were 925 radioisotope shipments during December, compared with 815 last month. During 1952, 10,691 radioisotope shipments were made, compared with 9491 in 1951 (p. 23).

REACTOR OPERATIONS DEPARTMENT

OPERATING DATA

	DECEMBER 1952	YEAR 1952	YEAR 1951
ORNL Graphite Reactor			
Reactor power			
Total accumulated (kwhr)	2,569,688	28,995,986	29,997,102
Average kw/operating hr	3823.27	3684.83	3806.70
Average kw/24-hr day	3453.88	3301.00	3424.30
Lost time (%)	9.66	10.42	10.00
Excess reactivity (inhr)	40		
Slugs discharged	80	42,128	3544
Slugs charged	797	43,521	3657
Product made (g)	93.98	1058.25	1094.78
Product discharged (g)	1.43	6245.80	416.24
Low-Intensity Test Reactor			
Reactor power			
Total accumulated (kwhr)	1,014,646	9,399,538	1,339,589
Average kw/operating hr	1500.0	1287.0	647.8
Average kw/24-hr day	1364.0	1070.0	473.0
Lost time (%)	9.1	16.8	27.0
Position of No. 2 shim rod (in. out)	24.928*		

*This corresponds to approximately 1% excess reactivity.

REACTOR OPERATIONS

ORNL Graphite Reactor

A ruptured slug was detected in channel 1869 by visual inspection and was discharged without difficulty. The unbonded slug had been irradiated for I^{131} production and had been in the reactor for 119 days at a maximum temperature of approximately 255°C.

On December 29, 717 additional slugs were added to the reactor to offset the reactivity loss resulting from insertion of additional experimental equipment in the reactor, principally to offset the liquid-nitrogen-filled Dewar being inserted in hole 10. The loading was made by increasing the length of the rows in zones 3 and 4 by two slugs each and in zones 5 and 6 by five slugs each; this gave slightly over 50 inhr increase in reactivity. The excess reactivity at the end of the month was approximately 40 inhours.

Summary of Operations During 1952

Operation of the ORNL graphite reactor was normal during 1952 except that the large amount of material that was irradiated caused a shortage of reactivity and occasionally limited the power; consequently, the average power for the year was lower than in 1951.

There were 13 slug ruptures in 1952, compared with 15 in 1951; five were bonded slugs and eight

were unbonded slugs. Two of the unbonded-slug ruptures should be discounted because they were attributed to a low-melting aluminum solder used to fasten thermocouples to the end of the slugs. The number of slug ruptures prior to 1951 were: 13 in 1950, 14 in 1949, 14 in 1948, and 13 in 1947.

The maximum metal temperature of the reactor was increased from 245 to 275°C on May 14 and was further increased to 300°C on September 30. It is believed that unbonded slugs would have ruptured badly at these temperatures, since previous experience indicated that higher temperatures increased frequency of rupture.

Table 1 is a list of pertinent data on slug ruptures that occurred in 1952.

At the beginning of the year, bonded-slug production was at a standstill because of the large number of blisters and ruptures encountered when slugs were tested at 400°C for one week. A new process that was developed by the Metallurgy Division has apparently remedied the defects of the old process, since very few blisters or ruptures occur when slugs are tested at this temperature. The procedure is a modification of the lead-dip process by addition of a layer of molten flux above the aluminum-silicon alloy.

In February, bonded-slug production was resumed at Y-12, and the first bonded slugs were charged into the reactor on March 31. By the end of April, approximately 9000 bonded slugs had been charged,

TABLE 1. SLUG RUPTURES DURING 1952

NUMBER	CHANNEL	DATE DISCOVERED	DAYS IN REACTOR	APPROXIMATE TEMPERATURE (°C)	POSITION*	REMARKS
			Bonded			
100	1661	6/16	63	225		Lot 127 (beta phase)
103	1568	8/11	132	230	7	Lot 113 (alpha phase)
104	1372	10/13	189	250	19	Lot 118 (87% beta phase)
105	1871	10/13	195	250	21	Lot 110 (alpha phase)
106	1163	11/10	203	225	20	Lot 130 (beta phase)
			Unbonded			
95	1370	1/12	16	240		Low-melting solder on thermocouple slug
96	1274	1/15	15	180	19	Caused by solder on thermocouple slug
97	1775	1/28	2649	220		Eight slugs ruptured
98	1364	3/17	2744	220	12	Three slugs ruptured
99	1266	4/7	96	210		In thermocouple row
101	1556	8/4	2900 88	125 145		Found while recharging
102	1256	8/11	2936 88	100 120		Found while discharging
107	1869	12/8	119	255	3	Iodine slug row

*Determined by counting from west end of row.

and the production schedule for bonded slugs was decreased from 62,000 to 53,000. Approximately 16,000 bonded slugs had been charged to the reactor, completely filling the central zone, by the end of May.

The first bonded slug ruptured on June 16 (channel 1661), and the slug was found to be completely oxidized, which was unusual for a ruptured slug; none of the bonded slugs that ruptured subsequently have oxidized to any great extent. At the end of June, approximately 26,000 bonded slugs had been loaded into the reactor, and charging was stopped when it was discovered at Y-12 that beta transformation was not complete in a considerable portion of the slugs being produced. The slugs not in the reactor were returned to Y-12 for testing and were separated into lots of beta-transformed, partially transformed, and alpha slugs. It was possible to locate at least a few slugs of nearly every lot that had been produced and from this, to determine what fraction had been beta transformed.

Of the approximately 26,000 slugs charged by the end of June, some lots contained an appreciable number of alpha slugs, and it was considered desirable to study the effect of reactor temperatures on such slugs. Therefore, the Metallurgy Division set up a test in which the slug temperature was cycled from 350 to 50°C, and it was found that the jackets of some slugs increased considerably in length. In a few instances, the increase was as much as ½ in. after approximately 430 cycles; however, the uranium did not grow a similar amount, and this resulted in an empty space in the end of the can. To study the effect of reactor irradiation on such slugs, representative lots are occasionally discharged and individual slugs are measured in the canal. Also, it has been found possible to measure the length of slug rows while they are in the reactor. A number of rows have been measured and will be remeasured from time to time to detect any growth that might occur.

All slugs charged after July have been segregated into lots of beta-transformed, partially transformed, and alpha material, and records of the material charged in each row have been compiled. The reactor was completely charged with bonded slugs in September, except for a few slugs that were being irradiated for radioiodine; production of slugs at Y-12 was completed in November. Of the approximately 53,000 slugs accepted, 42,074 are in the reactor; this leaves 10,793 bonded and 1440 unbonded slugs in the vault. In addition, there are 175 unbonded slugs for radioiodine production, 21 bonded donut slugs, and 15 experimental bonded slugs in the reactor. There are 31 spare bonded donut slugs in the vault.

Reactivity of the ORNL graphite reactor had to be increased twice during the year. On August 25, 536 slugs were added to the reactor loading by increasing the rows in zones 1 through 6 by two slugs each, which made the total at that time 41,531, plus 21 donut slugs and 15 experimental slugs in channel 1866. On December 29, 717 additional slugs were added to the reactor by increasing the length of slug rows in zones 3 and 4 by two slugs each, and in zones 5 and 6 by five slugs each. Reactivity gains were approximately 50 inhr after each increase in loading.

A number of tantalum batches was irradiated for the Army Chemical Center, as shown in Table 2.

The 540 tantalum slugs now in the reactor were originally scheduled to be removed on September 29, but the Army Chemical Center indicated that they would be unable to use the tantalum at that time and wished to irradiate 69 other slugs to a high specific activity. It would have been impossible to do this without discharging the 540 slugs, and this material, if discharged, would have become worthless. Since its value was estimated at approximately $20,000, a decision was made to attempt to keep the tantalum in the reactor and to have the 69 slugs irradiated at some other location. The Army Chemical Center made arrangements to accomplish this at Brookhaven National Laboratory.

Several sources of Co^{60} were loaded; these included a 1000-curie source for the Los Angeles Tumor Institute, a 3400-curie source for KAPL, a 1000-curie source for an ORNL division, and a 1250-curie source for the ORINS Medical Division. Two of these sources were sealed, and the outside of the source container was cleaned so that no contamination could be spread by the source. One was sealed in the canal, and the other was sealed in one of the cells of the Solid State Division.

Cobalt stored in the canal was found to be a major source of contamination of the canal water. It was possible to isolate this contamination somewhat by building a wooden dam in the canal, with all the cobalt kept on the downstream side. After

TABLE 2. DESCRIPTION OF TANTALUM BATCHES IRRADIATED FOR ARMY CHEMICAL CENTER

DATE IN	NUMBER OF SLUGS	SLUG WEIGHT (lb)	DATE OUT	AVERAGE SPECIFIC ACTIVITY (curies/lb)	DATE SHIPPED	POSITION
3/10	30	1	3/31	30	4/30	Hole A, channel 1964
3/31	280	2	4/28	3	4/30	Peripheral
4/28	280	2	6/30	8	9/4	Peripheral
5/5	6	0.4 to 1.4	5/12	–	5/14	Central and peripheral
6/30	270	1	Still in reactor			Peripheral
7/7	270	1	Still in reactor			Peripheral
7/21	3	1	7/28	10	7/29	Hole B
8/27	30	1	9/29	30	10/9	Holes A and C

this had been done, the canal water above the dam stayed much cleaner, and the general radiation background was lowered appreciably. In March, carbon tetrachloride was put over the cobalt in an attempt to prevent further corrosion by the canal water. By May, it became apparent that the carbon tetrachloride, probably because of its decomposition, was accelerating the corrosion rate of the cobalt.

The ion-exchange demineralizer in the canal has continued to function satisfactorily, usually reducing the radioactivity in the effluent water from 10,000 to 20,000 beta counts/min/ml to about 300.

Liquid hydrogen was produced throughout the year as requested by research personnel; production averaged about 8 liters per month. Costs were accumulated and it was found that the average cost including labor, material, and overhead was approximately $24.00 per liter (labor, $8.00; material, $10.50; overhead, $5.50 per liter).

Both mattress plates were changed; the north plate on November 24 and the south plate on December 1. The north plate was in considerably better condition than the south plate, which had several holes in it. The plates had been changed previously on April 21 and April 14, 1952, respectively.

Low-Intensity Test Reactor

The operation of the LITR was normal during the month.

A study is being made of the LITR shielding, and it appears that additional shielding will be necessary on the exit water line, the seal tank, and several places around the reactor shield in the event of any major increase in power level.

To facilitate the return of spent fuel elements to Arco for separation, a fuel-element carrier has been designed, and the shell is being fabricated by an outside machine shop at a cost of about $3700.00. The shell will be filled with surplus scrap lead available at ORNL.

Summary of Operations During 1952

The LITR operated very satisfactorily during 1952. Several special experiments were performed with the reactor, and these, together with alterations made to increase the power, resulted in a slightly higher down-time figure than would normally be expected. In February and March, new water lines, pumps, and a heat exchanger were installed. In May, there were two water-drop tests and two days of training for the Reactor School students; in July and August, the reactor was shut down for almost two weeks in preparation for a boiling test.

In February, new 8-in. water lines were installed to replace the old 3-in. lines. A larger pump and a heat exchanger were also installed, which permitted the power of the LITR to be increased from 800 to 1500 kw on April 24. One of the most difficult jobs was to increase the exit water lines inside the reactor tank; this was done by installing two 6-in. lines, each of which had four 2-in. lines extending through the A-piece holes to a manifold at the bottom. The new lines were very satisfactory, since the pressure was only about 2 psi in the top of the reactor tank after the installation; previously, the pressure at this point was about 10 psi. In addition, the flow rate was increased from 300 to 1200 gpm.

Since the new exit water lines provided for collection of the water immediately after passage through the fuel elements, there was an increase in the amount of N^{16} (7-s half life) that came out of the reactor tank; consequently, the exit water line and the seal tank had to be shielded early in March.

The pumps, valves, and fittings were made of steel, whereas the new lines were made of aluminum. Where possible, the steel parts were painted with Amercoat, but this was not successful, since considerable paint came off and caused radioactivity in the circulating water to be increased during March and April. A strainer was installed in the water line, which caught most of this paint, and the radioactivity soon decreased.

The oscillations of the reactor power noted at the end of 1951 were traced to air that was being sucked in by the circulating pump. After installation of the new pumps, the oscillations stopped.

In May, two water-drop tests were made at 1000 and 1250 kw, respectively. In order to permit safe operation at higher power levels, it was decided to install an auxiliary water tank that would spray water over the fuel elements for a considerable time in case of a sudden loss of water from the reactor tank. Complete installation of the auxiliary tank has been delayed several months because of other work having higher priority.

To conserve plant water, a fluid cooler designed to replace plant-water (used to cool the LITR circulating water that was passed through a heat exchanger) with air cooling was purchased from the Trane Co. Operation of the fluid cooler was begun on November 25 and appears to be very satisfactory. With both fans operating at full speed, the fluid cooler gives a temperature differential between the air temperature and the exit water temperature of about 25°F while operating at a power of 1500 kw and a flow of 1200 gpm.

Corrosion in the reactor appears to be reasonably low; however, corrosion of the steel tank appears to be somewhat higher than was previously indicated by water analyses. On samples of SAE 1030 steel placed in the reactor tank, rust forms in a thick adherent coat over the exposed surfaces. Since the Amercoat paint on the steel tank walls of the reactor can be seen to be failing in numerous spots, it is assumed that the steel tanks are corroding at about the same rate as that indicated by these samples. The rates of corrosion on the samples were measured by the ORNL Corrosion group of the Reactor Experimental Engineering Division, and were found to be initially about 8 mpy with a steady-state rate of about 2.6 mpy. Some pitting was also noted, and this may be more serious. Several experiments on the reduction of corrosion are being considered.

Experimental facilities were increased in July by adding a third tube in the fourth beryllium row adjacent to the fuel. This tube, approximately 2 in. in diameter, fits into a hollow beryllium piece located in position C-42. A flexible metal tube extends from the top of the upper grid around the spider and out the southwest diffuser flange. In May, hole V-2, which extends at an angle down to the outside of the reactor tank, was equipped with a sliding door and a beam catcher so that samples could be installed and removed while the reactor is in operation. The neutron flux in this hole is quite low, and the hole is being used for activation-analysis irradiations. Pneumatic tubes were installed in August, which made short irradiations possible in the LITR.

A boiling test was made to determine the stability of the reactor while the water in the lattice was boiling. The reactor was started up, with the circulation stopped, on August 4. Boiling was found to be a fairly stable condition, with the power fluctuating over a range of several hundred kilowatts as boiling in the lattice alternately stopped and started. Boiling caused steam bubbles in the lattice, which decreased reactivity automatically and limited the reactor power.

FILTER HOUSE

Table 3 shows a comparison of the pressure drop across the exit air filters last month with the

pressure drop this month, one year ago, and that experienced when all filters were clean.

Summary of Operations During 1952

The filter house operated satisfactorily during 1952 without a single filter change. The last filter change was in the spring of 1951 when the CWS No. 6 filters were all renewed.

The following is a list of the previous filter changes:

Cell No. 1	AAF	5/8/50
	CWS No. 6	5/17/51
Cell No. 2	AAF	12/18/49
	CWS No. 6	5/25/51
Cell No. 3	AAF	10/23/50
	CWS No. 6	5/3/51
Cell No. 4	AAF	4/26/49
	CWS No. 6	5/10/51

At the current rate of increase in pressure drop across the house, it is expected that the AAF filter in cell No. 2 will be changed in May 1953, and that the AAF filter in cell No. 4 will be changed in November 1953. No changes of CWS No. 6 filters are anticipated during 1953.

FAN HOUSE

Both fans operated normally throughout December.

Summary of Operations During 1952

The No. 3 fan operated without difficulties throughout the year. The bearings were rolled out, and the bearings and shaft journals were inspected on August 18. Both appeared to be in excellent condition.

In May 1951, during a routine inspection of the No. 2 fan bearings, two horizontal lines of pitting, 180 deg apart, were noted on the shaft journal of this fan. The pitting was thought to have been caused by inferior oil. Therefore, the oil was changed, and the fan ran until November 1951 without further evidence of pitting; at this time a faint line was noticed in the outboard bearing 90 deg from the other lines.

On August 25, the bearings were found to be badly wiped, and the shaft was slightly scarred and badly pitted. The pitting consisted of four horizontal lines across the journal 90 deg apart.

The bearings were scraped back into shape and reinstalled. Since that time, five inspections have failed to show further damage to the shaft.

On October 20, thermocouples were installed in the steel-babbit housing of the bearings; these thermocouples should reflect a temperature increase in the bearings more rapidly than that indicated by the oil temperature and should give an immediate indication of a bearing failure.

RADIOISOTOPES

At the end of December, there were 381 cans of target material in stringers 13, 14, and 16, compared with 370 cans of target material in these stringers at the end of November.

Table 4 shows a comparison of the radioisotope and research samples charged into the ORNL graphite reactor during December with those handled in November.

Summary for 1952

Table 5 is a comparison of the radioisotope and research samples charged into the reactor during 1952 with those handled in 1951.

WATER-DEMINERALIZATION BUILDING

Table 6 shows water-treatment data for December, as compared with those for the years 1952 and 1951.

TABLE 3. PRESSURE-DROP DATA

DATE	PRESSURE DROP (in. water gage)		
	Glass Wool	CWS No. 6	Total Across House
12/31/52	3.7	2.4	7.3
11/30/52	3.6	2.3	7.1
12/31/51	3.2	1.6	6.1
Clean filters	1.1	1.3	3.3

TABLE 4. RADIOISOTOPE AND RESEARCH SAMPLES HANDLED IN NOVEMBER AND DECEMBER

	DECEMBER 1952		NOVEMBER 1952	
	Research	Radioisotope	Research	Radioisotope
Stringers 13, 14, and 16	15	166	12	132
Hole 22	55	4	68	1
All other holes	13	28	1	26
Total by groups	83	198	81	159
Total for month	281		240	

TABLE 5. RADIOISOTOPE AND RESEARCH SAMPLES HANDLED DURING 1951 AND 1952

	1952		1951	
	Research	Radioisotope	Research	Radioisotope
Stringers 13, 14, and 16	246	1872	169	1765
Hole 22	677	46	763	50
All other holes	78	270	75	297
Total by groups	1001	2188	1007	2112
Total for year	3189		3119	

TABLE 6. WATER DEMINERALIZED AND DEGASIFIED

	WATER TREATED (gal)		
	December 1952	Year 1952	Year 1951
Demineralized	403,086	4,747,664	6,750,085
Degasified	0	323,120	492,121

Summary of Operations During 1952

Resins in the ion-exchange columns of the Water-demineralization Building were changed. Rohm and Haas resins IR112 and IRA 410 were substituted for Permutit resins ZK and DA. The new resins made it possible to produce water with specific resistances between 5×10^5 and 10^6 ohms, as compared with a maximum resistance of about 100,000 ohms previously. The new resins also eliminated the need for vacuum deaeration or degasification to remove CO_2.

CHEMICAL SEPARATIONS AND RADIOISOTOPE DEVELOPMENT DEPARTMENTS

RADIOISOTOPES

Iodine (I^{131} – 8 d)

Seventy ORNL slugs were processed and 39,363 mc of iodine was shipped.

The yields this month were, in general, lower than usual. Some of the low yields were caused by either a color or a precipitate that appeared in the product and necessitated reprocessing; some low yields cannot be explained.

Attempts to increase the product yield by substituting nitrogen for air in the first distillation operation appeared to accomplish little, if any, improvement.

A reduction in the nitric acid concentration during the dissolving step was also tried but gave no conclusive results.

Summary of Operations During 1952

Table 7 gives a comparison of the amount of material processed and the product shipped during the last three years. The small increase in the amount of product produced per pound of uranium in 1952 can be attributed to the greater number of Hanford-irradiated slugs processed.

The performance of the equipment during 1952 was as good as could be expected with the equipment operated at so much above its design capacity. Only one major decontamination of the extraction cell was required to remove a damaged tantalum liner in the dissolver and to replace a leaking gasket on the same vessel. The glass purification equipment was also completely overhauled once during the year.

The rotation of operating personnel, as mentioned in last year's summary, was continued throughout the year in order to keep the radiation exposures below tolerance levels. This inefficient method of operation will be eliminated when the new radioiodine plant, now under construction, is completed.

Iodine Development Work

All underground piping was completed, and concrete was poured for the base of the cells and for the filter box in Building 3028. Fabrication of the stainless steel cell liners was completed.

Changes suggested by the Chemical Separations Department in the evaporator and dissolver vessel design and in a portion of the piping design were given to the Engineering Department for incorporation in revised drawings. A transfer line from cell 2 to Building 4501 cells was added at the request of the Chemistry Division.

Additional experiments with the bellows-pump sample-transfer system proved that this system is unable to pump a suction head of 14 ft as required in the installation. A system utilizing a well-known type of sampler in which the sample is drawn through the sample bottle to a vacuum transfer tank was tested. Vacuum was provided by an air-jet evacuator. Automatic cutoff of the vacuum and venting was provided by probes in the transfer tank, which operated a three-way solenoid valve through an electronic relay. It was decided to use this system, and a full-scale test layout of the system was begun.

Design of the instrument console and associated instruments, and of the ion chambers, was completed by the Instrument Department.

Summary of Development During 1952

Final approval for the construction of the new I^{131} plant was received from the AEC in August,

TABLE 7. IODINE PROCESSED AND SHIPPED

YEAR	ORNL SLUGS	HANFORD SLUGS	URANIUM (lb)	MILLICURIES SHIPPED	MILLICURIES SHIPPED PER POUND OF URANIUM USED
1952	644	10, 4-in. & 2, 8-in.	1696	488,231	288
1951	651	5, 4-in.	1693	467,544	276
1950	396	12, 4-in.	1066	310,698	291

and construction was started in November by an outside contractor, Johnson and Willard of Knoxville, on a cost-plus-fixed-fee basis. Some subcontracts, such as those for external piping, were let on a lump-sum basis. The fabrication of most of the equipment is being done in outside shops, but installation of equipment, internal piping, and instruments will be done by ORNL forces. It is expected that this unit will be ready for testing operations by June 1953.

Little further developmental work was done on the chemical process during the year, since the design had been frozen for over a year. It was established, however, that oxygen could be added to the cold-water scrubbing tower to cut down hydrogen peroxide requirements and that a distillation fractionating column could be used for concentrating iodine in the final purification. Several steps in the chemistry of the final purification are still under investigation, but this work will not necessitate changes in the general design of the plant under construction.

Considerable attention was also given to the sampling of the process streams at various points for control purposes, and it was decided to combine facilities for sample receiving and analysis to reduce handling of radioactive material and to speed up operations.

Phosphorus (P^{32} – 14.3 d)

Twenty-two 2500-g cans of bombarded sulfur were processed and 10,013 mc of P^{32} was shipped.

A high loss of product was experienced in three runs because of the reprocessing made necessary by a precipitate appearing in the product when it was adjusted to a pH of 7 to 8. One run was almost completely lost when a sintered-glass filter disk broke under vacuum.

In last month's report it was explained that neodymium hydroxide in the presence of ammonium chloride as a buffer was used in the precipitation step to replace lanthanum. If, as anticipated, the precipitate was magnesium silicate, it was hoped that this change would reduce the precipitate at a pH of 7 to 8. It now appears that the neodymium hydroxide did not aid in the reduction of this precipitate in the final product, and its use has been discontinued.

Summary of Operations During 1952

Table 8 gives a comparison of the amount of material processed and the product shipped during the last three years.

An attempt was made during this year to keep a larger quantity of product on hand in order to satisfy emergency requests and to improve service to customers. This policy may account for the small decrease in the amount of product shipped per kilogram of sulfur processed.

The performance of equipment was usually satisfactory during the year. The greatest difficulty was the periodic reappearance of a precipitate when the pH of the product was adjusted to 7 to 8.

Summary of Development During 1952

The purification of target material, elemental sulfur, for P^{32} production was of principal interest during 1952. Two methods were tried: (1) extraction of impurities with weak nitric acid and water from molten sulfur in a pressure extractor, in a manner very similar to the extraction of P^{32} in the production process; and (2) passage of molten sulfur through a bed of magnesium oxide, followed by a fine filtration. The second method was chosen because of better removal of impurities, such as organic matter and SiO_2, and simplicity and speed of operation. Test amounts of sulfur were purified and used in the regular production process for part of the year. Although it was evident that the material was much cleaner

TABLE 8. SULFUR PROCESSED AND PHOSPHORUS SHIPPED

YEAR	SULFUR PROCESSED (kg)	MILLICURIES SHIPPED	MILLICURIES SHIPPED PER KILOGRAM OF SULFUR USED
1952	463	118,668	256
1951	406	107,187	264
1950	452	95,954	212

than formerly, some difficulty with the formation of precipitates in the final material was encountered during the latter part of the year. No clear connection could be established between the use of sulfur purified by the MgO method and the final precipitate, but it was decided to discontinue the use of the oxide pending further investigation.

Routine production of red phosphorus–bakelite plaques was continued, and 75 were prepared and calibrated for shipment during the year. New molds were designed and fabricated for making ¼- and ⅛-in.-dia rods of red phosphorus–bakelite; these rods are being offered as a new item in the radioisotope catalog.

Carbon (C^{14} – 5740 y)

An estimated 500 mc of C^{14} was produced during the month. The analysis is in progress.

Summary of Development During 1952

A total of 5521 millicuries of C^{14} was produced during 1952, with an average isotopic ratio of 10%. The highest isotopic ratio obtained in 1952 was 28%. In comparison, the average isotopic ratio obtained in 1951 was 11.5%, with a maximum of 25.3%.

No significant alterations were made in the process chemistry or equipment this year.

It was decided to discontinue recovery of beryllium as the basic carbonate until such time as a process for conversion to the nitride might be worked out. No further effort was expended on developing a method for producing beryllium nitride from waste beryllium, since it is believed that it will be possible to purchase especially purified beryllium nitride in pellet form suitable for irradiation.

Several special runs were made, including a separation on beryllium nitride, for the Solid State Division in connection with their work in determining the neutron spectrum in the LITR. Several batches of material were also prepared in the form of sodium carbonate solution rather than as solid barium carbonate.

The gas from one entire run was collected in evacuated bulbs, but time did not permit fractionation and analysis to determine the composition.

Fission Products – Summary of Development During 1952

Fission Products from Process Waste. A complete precipitation process for separating Cs^{137}, Sr^{90}, Ru^{106}, Ce^{144}, and the balance of the rare earths was designed, and the equipment was installed in Building 3515. Construction was prolonged by difficulties in obtaining several pieces of equipment, such as special Vycor vessels, and in finding valves that were suitable for work with this type of equipment. Essential features of this process are: (1) formation of an alkaline carbonate precipitate by using added calcium or barium carrier to precipitate alkaline earths, rare earths, and part of the ruthenium; (2) filtration to remove the precipitate and recover the filtrate for cesium separation; (3) removal of cesium from the filtrate by co-crystallization with ammonium alum; (4) separation of the alkaline-earth fraction by precipitation in 80% nitric acid; (5) distillation of ruthenium by oxidation with periodic acid; (6) precipitation of cerium as the iodate; (7) removal of other rare earths in the filtrate from the iodate precipitation.

A special batch of old W slugs was processed by the Scrup plant, especially to produce Purex concentrated waste containing Sr^{90} low in Sr^{89}. Enough of this material was processed by the end of the year to separate approximately 38 curies of crude Sr^{90} and approximately 100 curies of Cs^{137}. Difficulties were encountered because of an extraordinarily large amount of iron contamination in the feed material. This difficulty was temporarily overcome by the use of Versene Fe 3 Specific to complex the iron during the alkaline carbonate precipitation. The rare-earth fraction was removed and stored for further purification on the steam-heated ion-exchange column.

A significant chemical development was the removal of aluminum from the alum in the final stages of the cesium purification by precipitating aluminum hydroxide in a dense filterable form by hydrolysis of urea; the previous flow sheet called for removal of aluminum by ion exchange.

Notable new design features were: the inclusion of manometers inside a shielded enclosure where they are read by periscope, the use of Vycor vessels to withstand severely corrosive conditions, and the installation of all discontinuous lines from the operating panel to process vessels to prevent blow-backs into an unshielded area.

The concentrated waste from all the metal processed in the Scrup program is also available for processing in the fission-product recovery unit. Cesium-137 will be recovered, not only from Purex waste, but also from tailings of Redox waste previously processed by the Chemistry Division.

Fission Products from Dissolved Metal. Operations in the fission-product cell in Building 3026 for the separation of fission products from uranium metal dissolved in the I^{131} equipment are now in their fifth year. A small, batch-type TBP extractor of very simple design was used extensively for the first time this year and proved to be the most useful single piece of equipment. By using this extractor, fission-product mixtures, with sufficiently low levels of uranium and plutonium concentrations, were made that could be offered for sale in the radioisotope program. The TBP extractor eliminated the use of the large ion-exchange columns previously used to separate uranium and plutonium fission products. The obsolete pieces of equipment will be removed in the coming year, and larger batch TBP extractors will be added. It is planned to install improved process equipment in the Building 3028 fission-product cell that adjoins the new I^{131} plant.

Special slugs from Hanford were processed mainly to separate Sr^{90}; virtually all Sr^{90} production this year was from the Building 3026 unit. Fractions necessary for the production of Y^{91}, Pm^{147}, Nd^{147}, Pr^{143}, $Ce^{141,144}$, Sr^{89}, Ba^{140}, $Ru^{103,106}$, and Zr^{95}-Nb^{95} were also separated routinely. The silica-gel column for separation of zirconium-niobium was operated without difficulty during the year; the largest batch separated was approximately 100 curies. Preliminary studies and calculations were made which indicated that a kilocurie amount could be separated on the same 1-liter column. Shielding (local plus space shielding) sufficiently heavy to allow production of such amounts will be included in Building 3028 facilities. Many samples of UNH solution were removed for various ORNL groups during the year; this is the only facility in the Laboratory where hot UNH can be easily obtained.

Purification of Fission Products. Improvements were made in the processes and equipment for purifying fission products. The principle of separation of strategic pieces of equipment was followed, and provision was made for processing crude fission-product fractions of widely varying composition; these would originate either from operations on waste streams or from fractions separated from metal dissolved especially for fission-product production.

Mixed fission products or fractions were sent to a special preparation cubicle in Building 3030 where the last traces of plutonium could be removed by another small TBP extractor, much of the organic material destroyed by oxidation with fuming HNO_3, and the excess acid evaporated off. These preparations, which were in the proper chemical condition to be adsorbed on an ion-exchange column, were then sent to the fractionating columns in Building 3028. These columns are steam-jacketed ion-exchange columns, ½ in. in diameter and 8 ft in length, and are usually loaded with Dowex-50 cation-exchange resin to a height of about 4 feet. The columns are supported in the steel work, and the lower ends extend into the top of a cell containing remotely controlled equipment for measuring and recording the radiation of the effluents and for separating and handling the various fractions. Eluting solutions are degassed and fed into the columns on the third floor of the structure; the necessary pressure is maintained by reservoirs on the fourth floor. The entire column assembly totals 24 ft in height.

Two columns were in operation by the end of the year: one primarily for short-lived fission products and the other for long-lived fission products. Space is available for two more similar columns. These columns operated extraordinarily well; data from some fractionations indicated that from 800 to 1000 theoretical plates could be obtained.

Europium-155 and Sm^{151} were separated from fractions originally obtained from old Redox waste. Also, relatively large quantities of Pm^{147} were purified and permitted a large reduction to be made in the price of this isotope. Chemical development work was chiefly concerned with the separation of Am^{241} from Pm^{147}, separation of Sm^{151} and Eu^{155} by oxidation-reduction precipitation processes, and final finishing of citrate fractions by direct incineration instead of metathesis on small ion-exchange columns. Equipment was designed to carry out this last process and will be installed during 1953.

Cesium purification equipment was included in the main precipitation process plant, which freed a relatively high-level processing cell in Building 3030 for Sr^{90} purification. A significant chemical process development in Sr^{90} purification was the precipitation of carrier Ba^{++} as the chloride in

cold 13.5 *N* HCl by using HCl gas to achieve high concentrations; this replaced the HCl-ether mixture formerly used in this step and eliminated the introduction of organic matter and the explosion hazard. The mechanism of the carry-through of ion-exchange resin residual organic matter in crude Sr^{90} fractions was established: the sulfate group retained on the partially degenerated resin provides a linkage with alkaline earths. The linkage was broken by attacking the residue with concentrated hydriodic acid, which destroyed the sulfate group. In this manner, the carry-through of organic matter was reduced.

Kilocurie Source Loading. A process for concentrating 1000 curies of Cs^{137}, pressing it into pellet form, and sealing it into a container was developed. It was decided to install the process in the unused radium-beryllium loading cell in Building 3013 to take advantage of existing facilities. Seventy-five engineering drawings were made by the Engineering Department, and most of the equipment was fabricated by outside vendors. All except three pieces were received by the end of the year. Final assembly and testing will extend through the summer of 1953.

Cesium sulfate was selected as the most suitable compound to use for source fabrication, and the details of conversion from the hydrosulfate to the normal sulfate by heating at 900°C were worked out.

Extra shielding (layers of barytes concrete and lead) was laid on top of the cell in Building 3013, equipment not needed was cleared out, and all remaining equipment was inspected and repaired.

Tritium (H^3 – 12.4 y)

Experiments are in progress to determine the optimum conditions for absorbing tritium in thin films of zirconium. Ordinary hydrogen is being used in this work.

Tritium and Helium – Summary of Development During 1952

Several improvements were made in the handling and packaging of tritium. An all-metal system, in which tritium is stored in uranium metal as the tritide, was designed and constructed. When needed, H^3 is removed by heating the traps and pumping it out into the dispensing system. This procedure also allows the He^3 daughter to be separated and therefore obviates the palladium valve formerly used for this purpose. A micro gas-density balance was added to the system to allow an analysis of the gases (particularly in reference to the hydrogen content) when required. Material balances made on the system were 100% with a probable error of ±2%.

When Zr-H^3 targets were first made, they were usually so-called "thick" targets (0.001 to 0.003 in. of zirconium) on tungsten backing. Because of considerable demand for thin targets (100 to 300 $\mu g/cm^2$ of zirconium), a machine for evaporating zirconium metal onto various backing materials was purchased, and the techniques for making thin and medium thick (1 to 3 mg/cm^2 of zirconium) targets were worked out. A microresistance type of heater was made for use during the absorption of H^3. Most of the latter part of the year was spent in developing these techniques, and requests for these special targets should be handled routinely during the coming year.

Quantities of He^3 were received from another site, and this gas was packaged for shipment on a routine basis. Special purifications were made on He^3 for the Physics Division by careful fractionation on charcoal columns operating at the temperature of liquid nitrogen. An appreciable demand for ultrapure He^3 is foreseen; therefore, equipment has been designed and is being fabricated for installation as a complete system for this purpose.

Strontium Purification

The strontium purification equipment was improved as follows: (1) modification of the $BaCl_2$ precipitator to use HCl gas under a slight pressure, instead of under vacuum, to increase HCl solubility; (2) installation of a settling vessel to separate crud from incoming Sr^{90} crude; (3) installation of equipment to flush material from the top of the 83% HNO_3 filter back to the main precipitation vessel; (4) installation of a jet to send the contents of the cell catch-pan to the hot waste system, with only emergency overflow to the semi-hot waste.

The first run of Sr^{90} crude from the precipitation process is in progress.

Experiments on the solubility of $BaCl_2$ in very concentrated HCl are being continued.

Sr^{90} Source Fabrication – Summary of Development During 1952.

Several types of stainless steel source holders for Sr^{90} were fabricated and tested. The most

satisfactory type was a 1-in.-dia holder with a 1-mil-thick stainless steel window soldered over the top, under a retaining ring. The sources were assembled and soldered by remote control, pressure tested, and cleaned to a smear count of less than 100 cpm. Several cements and low-melting ceramics were investigated as agents for sealing activity into the internal cavity of the source, but no really satisfactory material was found. A 100-mc source was fabricated and sent to the Bureau of Standards for calibration. Fifteen sources containing a total of 13,020 mc of Sr^{90} were made; the largest individual sources contained 1 curie of activity.

Precipitation Process, Building 3515

Revisions to equipment in cell 2 were completed, and concrete block shielding was installed in the doorway. Processing of radioactive feed material is in progress.

Purex waste in tank 15, which was separated from uranium and plutonium in the Scrup plant especially to provide fission-product feed containing Sr^{90} with low Sr^{89} content, is being processed first, mainly to separate Sr^{90} and Cs^{137}. Seven runs have been completed. A very large amount of Fe^{+++} contamination present in tank 19 feed causes great interference in the fission-product recovery process because of the massive $Fe(OH)_3$ precipitate that comes down in the alkaline carbonate step. This difficulty has been partly corrected by complexing the Fe^{+++} with Versene Fe 3 Specific, although it is necessary to take a 20% loss of strontium and a 50% loss of rare earths because the Fe 3 Specific also complexes these elements to some extent. However, the process would be almost inoperable if it were not possible to at least partly sequester the relatively large amount of iron. No accurate estimate of the amount of Sr^{90} crude separated is yet available, but it is thought to be about 50 curies. Cesium-137 is being separated and accumulated in a large batch of alum crystals in the crystallizers; probably about 100 curies has been separated so far. The rare-earth fraction is being stored for separation and purification of Pm^{147}, Sm^{151}, and Eu^{155}. The Ce^{144} and Ru^{106} have decayed out of tank 19 material.

Results of the first runs indicate that a larger filter area is needed to filter the alkaline precipitate from precipitator No. 1. The feasibility of centrifuging this precipitate is also being investigated. It is proposed to install either a filter having a larger area or a centrifuge, after sufficient amounts of fission products have been separated in the present equipment.

More holdup tanks are also needed to effect a greater flexibility in processing. Tanks now used as both filter vessels and transfer vessels should be used only as filter vessels and should have an absolute minimum of transfer lines connected to them to avoid vacuum loss through leaks.

Design was begun on a bottom-loading carrier for Sr^{90} products, which will have lighter shielding than the present product carrier.

Source Assembly Station, Building 3013

The following equipment for the source assembly station has not yet been received from the vendors but delivery is expected as listed: calorimeter, January 30, 1953; solution transfer system, January 15, 1953; No. 2 soldering station, January 15, 1953. Difficulties in fabrication are delaying delivery of the calorimeter.

Installation of equipment that has been received will begin January 5, 1953.

Gamma Source Loading – Summary of Development During 1952

Two types of small stainless steel sources to hold evaporated gamma emitters, such as Cs^{137}, in amounts up to 10 curies were designed. One type is made so that the evaporated material can be worked down into a relatively small point source. Both types are closed with threaded plugs seating on thin lead gaskets; the threads are sealed with bakelite varnish.

A special motorized manipulator was set up in one cubicle of Building 3029 for loading this type of source.

Miscellaneous Source Fabrication – Summary of Development During 1952

The following sources were made this year.

Radioisotope	Number of Sources	Total Activity (mc)
Co^{60}	830	1,748,800
Cs^{137}	13	1,391
Nb^{95}	1	4
Ce^{144}	2	800
S^{35}	8	40
AgI^{131}	3	6,000
Ru^{106}	1	6

Ca^{45}	1	1
Sr^{90}	15	13,020

Detection of Co^{58} in Irradiated Nickel

An analysis by W. S. Lyon (Analytical Chemistry Division) of the cobalt removed from an ultrapure nickel target that was irradiated at Hanford revealed that Co^{58} was present, presumably because of the *n,p* reaction on Ni^{58}. This may be an economical method of producing a carrier-free isotope of cobalt that avoids the use of a cyclotron in which this isotope has been made heretofore. This possibility will be investigated further during the processing of the next nickel target.

Europium Target Material – Summary of Development During 1952

Europium is an interesting target material for producing concentrated hard gamma sources ($Eu^{152,154}$) because of its very large activation cross section (~875 barns). A study was made of the various europium concentrates offered on the market and the work necessary for purifying europium, as compared with direct purchase of highly purified Eu_2O_3. Sodium amalgam extractions were used for separating the samarium-europium group, and subsequent separation of europium was made by selective reduction to Eu^{++} and precipitation as the sulfate. Samarium contamination was reduced to less than 1% (limit of spectrographic method), and irradiation tests conducted in the LITR indicated that this quality of material will be satisfactory for source production. A brief investigation of the TBP extraction of rare earths from strong HNO_3 was made, but it was concluded that this method offered no advantage over precipitation methods. Work is in progress on the production of 1-mm-thick wafers of Eu_2O_3 by hydraulic pressing and subsequent sintering. Enough material to produce several kilocuries of $Eu^{152,154}$ will be ready for the MTR before the end of 1953.

Processed Radioisotope Production

Table 9 is a list of radioisotope product solutions that were prepared during December.

Production Summary for 1952

Radioisotope	Millicuries
Sb^{124}	1,720
As^{73}	4.479
As^{74}	7.089
Ba^{140}	820.45
Be^{7}	1,212.26
Cd^{115}	87
Ca^{45}	1,546.8
C^{14}	5,521
Ce^{141}	138
Ce^{144}	1,795.6

TABLE 9. RADIOISOTOPES PRODUCED DURING DECEMBER

PRODUCT SOLUTION	SOURCE	AMOUNT (mc)	SPECIFIC ACTIVITY (mc/g)
Calcium (Ca^{45} – 180 d)	Hanford reactors	12.0 88.6	25.1 22.9
Cobalt (Co^{60} – 5.2 y)	Hanford reactors	5620	5,278
Chromium (Cr^{51} – 26 d)	Hanford reactors	397	1,250
Potassium (K^{42} – 12.44 h)	LITR	278 313	154 184
Barium (Ba^{140} – 12.8 d)	Fission products, ORNL graphite reactor	216.4	Carrier-free
Ruthenium (Ru^{103} – 42 d)	Fission products, ORNL graphite reactor	240	73,000
Strontium (Sr^{89} – 53 d)	Fission products, ORNL graphite reactor	552	Carrier-free
Strontium (Sr^{90} – 25 y)	Fission products, Hanford reactors	965	Carrier-free

Cl^{36}	0.53
Cr^{51}	6,042.6
Co^{56}	0.315
Co^{57}	0.90
Co^{60}	5,655
Eu^{155}	141.5
Fe^{55}	937.2
Fe^{59}	658.6
Mn^{52}	1.98
Mn^{54}	40.34
Hg^{203}	8,502.5
Mixed fission products	13,343
Nd^{147}	266
Ni^{63}	140.4
Nb^{95}	763.4
K^{42}	828
Pr^{142}	1,280
Pm^{147}	2,805
Ru^{103}	335.2
Ru^{106}	70.5
Sc^{46}	2,600
Ag^{110}	263
Na^{22}	36.26
Sr^{85}	8.1
Sr^{89}	8,561
Sr^{90}	20,019
S^{35}	39,175
Tl^{204}	15,835
Sn^{113}	33.2
W^{187}	1,014
Y^{91}	3,494
Zr^{95}-Nb^{95}	10,167.3

Special Preparations

Twenty-two Co^{60} sources were loaded with a total of 150 curies. Two Cs^{137} sources, a 1-mc and a 50-mc, were made. A 1-mc Sn^{113} source was made for a customer interested in detecting Auger electrons. A 1-mc Ca^{45} source was made. Four ampoules were filled with a total of 291 curies of tritium. Iodine-131 was concentrated to 50 mc/ml for a special order. A special preparation of 12.98 mc of Ni^{63} was prepared as $NiSO_4 \cdot (NH_4)_2SO_4$.

Summary of Development During 1952

Many special preparations, such as the preparation of Ni^{63} as nickel ammonium sulfate for plating experiments at the Bureau of Standards and Be^7 as the sulfate in isotonic solution for biological work, were made for various customers. In general, there was a decrease in this type of work, as compared with last year, because of de-emphasis of such special work under AEC policy in favor of work done by commercial laboratories.

Hot Chemistry Cell – Summary of Development During 1952

Design and construction of a heavily shielded utility cell equipped with master-slave manipulators in Building 3029 were completed late in the year. The unit has been in use for two and one-half months, and techniques of operation are rapidly being established. Dry loading of metallic Co^{60} sources into complicated source holders and carriers has been accomplished with amounts up to 200 curies. It is estimated that 500 curies of Co^{60} can be handled with the present $ZnBr_2$ window, and this amount can be raised appreciably when a special glass window is installed. Simple equipment has been devised for use within the cell to open Hanford and ORNL graphite reactor irradiation cans, quartz ampoules, and LITR rabbits. Purification of many of the high specific activity radioisotope products is being done in this versatile cell.

Capryl Alcohol as an Extractant – Summary of Development During 1952

A brief investigation was made of the properties of capryl alcohol, which is to be used for the extraction of various elements of interest in radioisotope production work. A continuous extractor for handling relatively low volatility solvents such as capryl alcohol was designed for this purpose. Extractions were made from various strengths of HCl up to 12 *N*; the extraction was found to increase directly as the HCl concentration. The following elements were extracted: Co, Zn, Fe, and Cu. Not extracted were: Be, Ni, Mn, Ba, La, and Sr. However, the rate of extraction is too slow for the process to be practical for most separations.

Separations from Cyclotron Targets – Summary of Development During 1952

A satisfactory ion-exchange method was developed for separating carrier-free $Mn^{52,54}$ from chromium and the usual contaminants, V^{48}, Zn^{65}, and Cr^{51}.

The most satisfactory method found so far for separating carrier-free Na^{22} from magnesium targets is the precipitation of the bulk of the magnesium as the ammonium phosphate, followed by final separation on ion-exchange columns. However, this is

considered as only an interim method, and the search for better methods will continue.

The Y-12 cyclotron was used very effectively for producing Be^7 from lithium, and exploration work is in progress on the preparation of carrier-free $Co^{57,58}$ from nickel, low specific activity Na^{22} from normal sodium, and Zn^{65} from copper.

High Specific Activity Short-Lived Isotopes from the LITR – Summary of Development During 1952

Potassium and sodium carbonate samples were irradiated in special tubes in magnesium rabbits in the LITR, processed, and analyzed on a fast schedule to enable distribution of high specific activity K^{42} and Na^{24} in solution form. The total time required from reactor discharge to placement in the shipping container is about 3 hours. Chemical work is done in the hot chemistry cell.

Extraction of Iron with TBP – Summary of Development During 1952

Appreciable quantities of Fe^{+++} can be extracted from 10 *N* HNO_3 solutions with TBP, which leaves impurities such as Co^{60} in the raffinate. This method was adopted for purification of all $Fe^{55,59}$ preparations, except Fe^{59} made from enriched Fe^{58}, which is extracted with dichloro diethyl ether.

Underground Storage of Hot Material – Summary of Development During 1952

A 40-hole underground storage garden, which consists of 6-ft-long, 3-in.-dia stainless steel tubes buried in packed crushed rock and capped with moisture-tight lead shielded plugs, was built east of Building 3028. Eventually, all Co^{60} that has been removed from the aluminum cans and is therefore subject to corrosion will be segregated, calibrated, and placed in this underground garden. Highly radioactive liquid fission-product fractions can also be stored in this facility.

Miscellaneous Work

The radiation-darkened lenses were removed from the periscope in the building 3026 fission-product cell and were cleared by baking. The periscope is now in good working condition.

The Building 3026 fission-product cell is being decontaminated so that a larger TBP batch extractor may be installed.

An as-built drawing of the canal tongs is being made and is about 75% complete.

Considerable difficulty had been encountered by reactor department personnel in identifying samples removed from the LITR. Several methods of engraving were investigated to alleviate this trouble. A satisfactory solution seems to be the use of a Handie tool for engraving wider letters that would be more visible. It has also been found that degreasing of the cans before insertion in the LITR may be an aid in diminishing the darkening of the cans in the reactor.

Summary of Development During 1952

Corrosion tests were made on titanium metal in a mixture of dilute HNO_3 and molten sulfur at 140°C. After two months of exposure, there was no measurable corrosion of the titanium. Autoclave Engineers, Inc. was consulted about designing and fabricating an autoclave made of titanium for use in extracting P^{32} from sulfur.

Experiments were made on preventing decomposition of $ZnBr_2$ solution in high radiation fields by the inclusion of fine zinc granules. This procedure was successful, but the zinc must be placed at the sides of the window to prevent interference from the gas bubbles that are formed.

The specific activity of Ce^{144} separated from Redox waste was measured and found to be 1.1 g of cerium per kilocurie of Ce^{144}; the calculated value is 1.6 g of cerium per kilocurie.

Silver iodide (AgI^{131}) was prepared in special plastic tubes for rain-making experiments by the Air Force.

Various cements and paints were subjected to 10^9 r in a Co^{60} irradiator to determine their suitability for sealing small source closures and identification labels in hot storage. Pliobond, bakelite varnish, and Sauereisen No. 1 and No. 31 were found to be satisfactory cements; porcelain refrigerator enamel was chosen for marking hot storage bottles. One plastic, Boltaron, was also checked and found to exhibit little or no injury after a 10^9-r dose.

RADIOACTIVE-WASTE DISPOSAL

Corrosion coupons were installed in all accessible stainless steel monitoring tanks to determine the corrosion effects of wastes on these tanks. If the rates are found to be excessive, wastes may have to be neutralized while they are being collected in the monitoring tanks.

Extensive tests made to determine the source of underground water leakage into tank W-8 were unsuccessful. Further attempts to locate the leaks were temporarily abandoned because the leakage was not considered great enough to justify the expensive digging operations that would be required to continue the investigation.

A large leak was again found at the flange of the W-1 tank outlet valve. A new gasket was installed in this flange last month, but it failed to hold because the wrong size had been used.

In an effort to locate the leakage of activity to the retention pond, the following steps were taken:

1. Tanks W-1 and W-2 were thoroughly tested for leaks. It was found that tank W-1 does leak but not in sufficient quantity to cause the high retention-pond discharge. However, tank W-1 has been removed from service.
2. The outlet line from the W-1 and W-2 diversion box to tanks W-1 and W-2 was uncovered from the diversion box to the Y in the lines above the tanks; no leaks were found.
3. Test holes are being drilled in the area adjacent to the route of the hot waste line from Building 3019 to tank W-5, particularly where this line comes near the retention-pond line, since it seems probable by the analysis of retention-pond discharge that a leakage from this line may be finding its way into the retention-pond system.

Approximately 9600 gal of concentrated chemical waste from tank W-8, which contained 235 curies of activity, was moved to chemical waste pit No. 2. The total transferred to date is 42,600 gal bearing 953 curies of activity.

Twenty-two carboys of normal uranium waste were received from General Electric Co., Cincinnati, and were emptied into tank W-7.

Eight pots and two drums of waste were received from Argonne National Laboratory. Five pots were emptied into tank W-7, and three pots and two drums were emptied into tank W-5.

Three hundred gallons of chemical waste was received from K-25 and was emptied into tank W-5.

The hot off-gas system was shut down for 2 hr and the hood ventilation was shut down for 7 hr to permit tie-in of lines from the new Radioiodine Processing Building, 3028.

The thrust ball bearings on both hot off-gas blower shafts and the coupling on the steam turbine hot off-gas blower were replaced as the result of failures.

The hot off-gas system in Buildings 4500, 4501, and 4505 was placed in service on December 11. The air flow through the off-gas system, as measured by a velometer, is now about 650 cfm. The recording flowmeter at the stack is not operating correctly and will be repaired. Its erratic operation is partly due to water condensing in the instrument lines.

The hood and cell ventilation fans, motor, and turbine of the 4500 area were run this month for checking purposes; this system is now ready for operation.

The Cottrell precipitator was shut down for 32 hr this month to permit the installation of extension valve handles for washdown facilities through the new brick shielding wall and to perform the semi-annual inspection and cleanup of the insulators and control room.

Waste Discharged to White Oak Creek

A total of 89.9 curies of beta activity was discharged to White Oak Creek from the settling basin and the retention pond (see Table 10). Approximately 80% of the total came through the retention pond from a source that has not yet been located, and about 5% was discharged from the Radioisotope Waste Processing Building, 3515, as a result of operating difficulties. The remaining 15% may be considered a normal discharge.

TABLE 10. ACTIVITY DISCHARGED TO WHITE OAK CREEK

DISCHARGED FROM	DECEMBER 1952		NOVEMBER 1952	
	Gallons	Beta Curies	Gallons	Beta Curies
Settling basin	15,953,000	15.51	15,719,000	29.83
Retention pond	468,000	74.42	364,000	8.25
Total	16,421,000	89.93	16,083,000	38.08

Chemical-Waste Evaporator

The waste-evaporator operation had to be maintained at less than 200 gph to keep the entrainment of activity down to desirable levels. This rate of operation was adequate for keeping up with the low quantity of wastes received this month. Evaporator operation is shown in Table 11; waste-tank inventory is shown in Table 12.

Summary of Radioactive-Waste Disposal During 1952

The total volume of hot wastes received at the tank farm in 1952 was approximately 2.3% lower than that received in 1951 and 1% lower than in 1950. This slight decrease, accomplished in spite of the accelerated pace of the chemical processing programs at the Laboratory, was made possible through use of the telemetering system installed as a part of the monitoring tank system. The telemetering system, which was completed during the first half of the year, has been found extremely valuable in controlling excessive volumes discharged into the hot waste system. The hot wastes received at the tank farm are shown in Table 14.

The total volume of waste discharged from the settling basin and the retention pond into White Oak Creek was also reduced from approximately 25,000,000 gal/month at the beginning of the year to 16,000,000 gal/month at the end of the year. This was accomplished by rerouting much of the cooling water from equipment such as air conditioners, etc., from the process waste system to the storm sewer. The importance of this volume reduction is not yet evident, since this waste is not

TABLE 11. WASTE-EVAPORATOR OPERATION

MONTH	SOLUTION FED TO EVAPORATOR (gal)	CONCENTRATE TO W-6 (gal)	VOLUME REDUCTION	BETA CURIES TO EVAPORATOR	BETA CURIES TO SETTLING BASIN
December 1952	143,431	10,147	14.1:1	37,000*	0.63
November 1952	159,826	12,641	12.6:1	46,348	2.61

*Estimated to within 1,000 curies; analysis of sample from one run nat considered valid because it was in a slurry form.

Waste-Tank Inventory

TABLE 12. WASTE-TANK INVENTORY

TANKS	CAPACITY (gal)	FREE SPACE (gal)	
		December 1952	November 1952
Hot-Pilot-Plant Storage			
W-3, 13, 14, 15	48,500	30,400	30,500
Chemical-Waste Storage			
W-5	170,000	103,000	90,000
Evaporator-Concentrate Storage			
W-6, 8	340,000	60,500	58,000
Metal-Waste Storage			
W-4, 7, 9, 10	543,000	209,500	212,500

TABLE 13. BETA CURIES DISCHARGED DURING 1950, 1951, AND 1952

YEAR	BETA CURIES FROM SETTLING BASIN	BETA CURIES FROM RETENTION POND	TOTAL
1952	411	87	498
1951	169	3	172
1950	172	15	187

TABLE 14. WASTE-EVAPORATOR OPERATION FOR 1950, 1951, AND 1952

YEAR	WASTE RECEIVED (gal)	SOLUTION PROCESSED (gal)	BETA CURIES IN FEED	BETA CURIES DISCHARGED TO SETTLING BASIN	DECONTAMINATION FACTOR	AVERAGE RATE OF PROCESSING (gal/hr)
1952	2,178,000	2,258,155	217,474	23.77	9149	257
1951	2,229,000	2,474,254	57,285	7.24	7912	282
1950	2,200,000	2,481,207	22,631	7.56	2994	283

treated prior to disposal. It has become apparent during the past year, however, that the activity discharged by the process waste system is reaching serious proportions and that it will eventually be necessary to treat this waste. Preliminary studies involving treatment of these wastes have already been started.

Although the waste volumes have been satisfactorily lowered during the last year, the total activity discharged to White Oak Creek increased from 172 curies in 1951 to 498 curies in 1952, as shown in Table 13. The bulk of the activity discharged through the settling basin resulted from accidental spills into the process waste system. The system was intended for this purpose, and the only apparent difference between the accidental discharges in 1952 and those of previous years was that the solutions contained considerably more activity. This is borne out by the increase in the amount of activity handled by the waste evaporator as shown in Table 14.

Practically all the activity discharged through the retention pond occurred late in the year and may be accounted for by two leaks, one at the tank W-1 drain valve, which has been repaired, and the other not yet located. As noted in the first part of this report, an extensive search is now being made to locate this source of activity.

The first chemical waste pit, dug in 1951 to check the practicality of permanent disposal of chemical wastes by absorption into the soil, was evaluated by the Health Physics Division and found to be highly satisfactory. A permanent pit, located in a more desirable place from the standpoint of terrain and quality of soil, was dug in the spring of 1952. To date, approximately 42,600 gal of waste-evaporator concentrate has been transferred to the new pit. It is hoped that disposal of radioactive chemical wastes into these pits will solve the problem of permanent disposal of wastes at the Laboratory.

RaLa (Ba^{140} – 12.5 d)

The rebuilding of resin cubicle 300 progressed this month to a point where it could be temporarily connected to the panel-board service for testing. Several minor pipe leaks and inoperable instruments were found and repaired, and the tanks were recalibrated.

Two dummy runs were made through the cubicle while it was connected above ground. Some minor troubles were encountered in the first run but were easily corrected. The second run presented no operating difficulties whatsoever.

The product yields from the two runs were poor, as has been the usual experience in previous dummy

runs. To be sure that the low yields were not caused by a damaged product evaporator filter, the filter was tested with a standard precipitation procedure normally used in hot runs and was found to be in good condition. As in other dummy runs made in the past, there seems to be no explanation for the low yields.

Two new sample carriers have been received for resin cubicle 300 and are being aligned to the sampling equipment.

The process filter cubicle was partly reassembled by rewelding the side into the stainless steel liner and rebuilding the lead shield. The replacement and retesting of the filters and manifold valves will be done after resin cubicle 300 is placed into its shield.

The lead cubicle, which housed the old glass purification equipment that has not been in use for over two years, is being dismantled for salvage.

Summary of Operations During 1952

A total of 119,000 curies of product was shipped this year in five batches. The largest shipment contained approximately 28,000 curies, whereas the smallest contained 18,500 curies. The smallest batch was almost 10,000 curies greater than any batch made prior to 1952. The shipping schedules were usually satisfactory to Los Alamos, although on one occasion, which was, unfortunately, during a period when the need for the product was critical, a shipment was completely missed because of processing difficulties encountered by equipment failure.

The quality of the product was not completely satisfactory to Los Alamos. Although the material always appeared to be far cleaner than any product made in previous years in the old equipment, Los Alamos found that purification was required before the product could be used. There is evidence that at least a large portion of the impurities are organic that are suspected to be coming from the resin or the Versenate used in the process.

All the runs were made with the redesigned and rebuilt resin cubicle equipment,[1] in which the process used was modified to eliminate the erratic pH adjustment of the feed solution. The changes were a great improvement over the original process and equipment.

The rebuilding of the standby equipment, resin cubicle 300, which was supposed to have been held up until the first rebuilt cubicle was tested, was not started until late in October. Until that time, the future of the program at Los Alamos was very uncertain, and it was believed possible that the program might be discontinued before the equipment was put to use. As noted in the first section of this report, the cubicle is now essentially complete. No design changes were made since the first cubicle was rebuilt over a year ago, because it was believed impossible to make further improvements in the limited space available within the shield.

In producing the large batches, it became very obvious that the existing facilities, portions of which were designed for 500 curies and others for 10,000 curies, were highly overtaxed and that further alterations in an effort of permanentization would be impractical and wasteful. Besides the lack of reliability required in production equipment, which makes the present plant undesirable, it is doubtful that long-term operations can be continued without overexposure of personnel to radiation.

The decision governing the future of the Los Alamos program was expected last August, and it was hoped that if a long-range program was adopted, another decision to build permanent facilities would follow shortly. The status of this program at the end of the year is not clear, and it is hoped that the meeting now scheduled for January 6, 1953, in Washington, D. C., will help clarify this situation.

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[1] The history and need for the project was reviewed in *Operations Division Monthly Report for Month Ending December 31, 1951*, ORNL-1212, p. 24.

RADIOISOTOPE SALES DEPARTMENT

Radioisotope shipments made during December 1952 are compared with those made during November 1952 and December 1951 in Table 15. A breakdown according to separated and unseparated material (including totals for 1951, 1952, and for August 1946 through December 1952), and for project, nonproject, and foreign shipments is also shown.

HANFORD IRRADIATIONS

The following radioisotope samples, which were discharged on November 25, 1952, were received from Hanford on December 16.

Sample Number	Material	Number of Pieces
ORNL-144	Chromium metal	1
ORNL-164	Hafnium oxide	1
ORNL-165	Calcium carbonate	2

CYCLOTRON RADIOISOTOPES

Table 16 is a list of the orders now on hand for cyclotron radioisotopes.

Table 17 is a list of the number of radioisotope bombardments received and requested through December 1952.

Table 18 shows the quantity of radioisotopes that was cyclotron-processed and the number of shipments of radioisotopes made during December. Also shown are totals to date as divided into millicurie and service-irradiation amounts.

ACTIVATION ANALYSES

A total of 79 requests has been received for information concerning activation analyses. Twenty-four of these have developed into requests for analyses, 15 of which have been completed.

TABLE 15. RADIOISOTOPE SHIPMENTS

	DECEMBER 1952	NOVEMBER 1952	DECEMBER 1951	TOTAL SHIPMENTS 1951	TOTAL SHIPMENTS 1952	INCREASE (%)	AUGUST 1946 TO DECEMBER 1952, INCLUSIVE
Separated material	749	670	622				31,117
Unseparated material	176	165	127				8,492
Total	925	835	749				39,609
Nonproject	824	745	627	7661	9,318	+21	
Project	89	19	104	1652	1,129	−32	
Foreign	12	71	18	178	244	+4	
Total	925	835	749	9491	10,691	+13	

TABLE 16. CYCLOTRON-RADIOISOTOPE ORDERS

MATERIAL	AMOUNT (mc)	SERVICE IRRADIATIONS	STATUS
Na^{22}	30.5		Material process
Ti^{44}		1	Material has been requested
Co^{57}	1.0		Material has been requested
I^{125}	1.10		Material has been requested

TABLE 17. BOMBARDMENTS RECEIVED AND REQUESTED

MATERIAL	MASS. INSTITUTE OF TECHNOLOGY		UNIVERSITY OF CALIFORNIA		UNIVERSITY OF PITTSBURGH		WASHINGTON UNIVERSITY	
	Bombard-ments	Beam Hours	Bombard-ments	Beam Hours	Bombard-ments	Beam Hours	Bombard-ments	Beam Hours
Bombardments Received								
Be^{7}			1	47.20	15	342.00		
Na^{22}	2	190.00			14	705.25	4	300.00
Mg^{28}			2	19.80				
Mn^{52}					2	20.00		
Mn^{53}			1	8.00				
Mn^{54}	1	50.00	2	182.40			7	400.00
Fe^{54}			1	18.70				
Co^{57}	1	10.00					4	140.00
Fe^{59}			7	332.80	2	80,60	1	34.00
Zn^{65}	1	100.00	1	47.80				
Ga^{67}					11	77.75		
As^{73}			1	10.50	4	40.50		
As^{74}	1	5.00						
Sr^{85}	3	89.75			1	10.00		
Y^{88}	1	10.00						
I^{125}							2	60.00
Iridium							1	8.00
Molybdenum metal			1	10.00	2	15.70	5	50.40
Potassium chloride	1	1.00						
Sulfur					1	2.00		
Total received	11	455.75	17	677.20	52	1293.80	24	992.40
Bombardments Requested but not Received								
Ti^{44}			1	10.00				
Sb^{125}			1	100.00				
Total hours outstanding (not received or requested)		1044.25		712.80		206.20		507.60

TABLE 18. SHIPMENTS OF CYCLOTRON-PROCESSED RADIOISOTOPES

MATERIAL	NUMBER OF SHIPMENTS DECEMBER 1952	TOTAL PRODUCT (mc)		NO. OF SERVICE IRRADIATIONS	
		December 1952	To Date	December 1952	To Date
Bi_2O_7	1			1	1
Be^7			376.743		4
Be^9	4			4	10
Na^{22}			87.557		
Mn^{52}			10.991		
Mn^{53}					1
Mn^{54}			22.22		
$Fe^{55,59}$			63.64		4
Co^{57}			4.144		
Zn^{65}			43.5		4
Ga^{67}	4			4	43
As^{73}	3	2.02	6.780		
As^{74}					1
Sr^{85}			6		
Sr^{88}			25		
Sr^{90}					1
Mo^{95}					9
Pm^{146}	1			1	1
KCl					1
Iridium foil	1			1	1

SF MATERIAL CONTROL

One carload-lot shipment consisting of 140 irradiated uranium slugs for the Purex process and one lead pot loaded with miscellaneous radioisotopes was received from Hanford.

Thirty-five MTR fuel assemblies and seven control rods were shipped to Phillips Petroleum Co., Scoville, Idaho, during December. This brings the number shipped to date to 279 fuel assemblies and 46 control rods.

At the request of the AEC, 1158 kg of Purex process depleted uranium in the form of UNH was shipped to Harshaw Chemical Co., Cleveland, Ohio, for denitration.

An over-all material balance of the Scrup process is expected to be completed during January 1953.

After a considerable amount of study and surveying relative to starting a quality control program over scales and balances used at the Laboratory for weighing SF material, it is noted that authorization has been given the Research Shops to begin a systematic, periodic testing, checking and calibrating program on equipment used in measuring significant quantities of SF material. This program was instituted in conformity with recommendation No. 3, *USAEC SF Accountability Survey Report No. 35*, dated January 29, 1952.

In compliance with recommendation No. 2, *USAEC SF Accountability Survey Report No. 35*, the Laboratory's internal inventory report, Form X-333, was modified to provide space for data that support the entries and methods employed in determining material balances reported.

There were 25 receipts and 30 outgoing shipments of SF material during December, compared with 27 receipts and 26 outgoing shipments during November. Tables 19 and 20 are summaries of receipts and shipments for December.

In connection with loading and unloading carload-lot shipments at K-25, it is noted that in January 1952 the SF Office estimated that there would be approximately 25 inbound and 25 outbound cars. The actual number received and shipped during 1952 was 26 and 25, respectively. It is anticipated that an equivalent number will be received and shipped in 1953.

TABLE 19. SF MATERIALS RECEIVED

FROM	MATERIAL	NUMBER OF SHIPMENTS	AMOUNT (g)
Argonne National Laboratory, ANL	Depleted uranium	2	2,852.10
	Plutonium		1.16
Brookhaven National Laboratory, BNL	Depleted uranium	1	17.83
	Plutonium		0.008
Carbide and Carbon Chemicals Co., K-25, CCC	Normal uranium	1	308.00
	Depleted uranium	3	1,537.00
	Plutonium		0.025
Carbide and Carbon Chemicals Co., Y-12, CYT	Enriched uranium	4	3,299.41
	Normal uranium	2	14.45
General Electric Co., AGT	Enriched uranium	2	8.20
	Normal uranium	1	841.40
General Electric Co., HGE	Depleted uranium	1	248,930.00
	Plutonium		146.00
Los Alamos Scientific Laboratory, SFC	Depleted uranium	1	11.95
	Enriched uranium	1	0.18
National Lead Co. of Ohio, NLO	Normal uranium	1	899,950.00
North American Aviation, Inc., DNA	Normal uranium	1	1.00
Phillips Petroleum Co., MTI	Enriched uranium	3	841.59
U. S. Naval Radiological Defense Laboratory, NRD	Normal uranium	1	0.935

TABLE 20. SF MATERIALS SHIPPED

TO	MATERIAL	NUMBER OF SHIPMENTS	AMOUNT (g)
Brookhaven National Laboratory, BNL	Enriched uranium	1	10.80
Carbide and Carbon Chemicals Co., K-25, CCC	Depleted uranium	1	202.00
Carbide and Carbon Chemicals Co., Y-12, CYT	Enriched uranium	4	20.54
	U^{233}	3	0.82
	Depleted uranium	3	20.35
	Normal uranium	1	88.27
E. I. du Pont de Nemours & Co., SRD	Normal uranium	2	24,050.00
Harshaw Chemical Co., MHR	Depleted uranium	1	1,158,239.00
North American Aviation, Inc., DNA	Normal uranium	1	1.00
Phillips Petroleum Co., MTI	Enriched uranium	9	6,806.50
Sylvania Electric Products Co., SYL	Normal uranium	1	6,150.00
USAEC, Chalk River, EVG	Normal uranium	1	85.00
	Plutonium		0.05
USAEC, New York Operations Office, COL	Normal uranium	1	0.40
USAEC, Oak Ridge Operations Office, CPA	Enriched uranium	1	0.27

Progress during 1952 included actual adoption of provisions to initiate, where feasible, the major recommendations of *AEC Survey Report No. 35.* In addition, as a result of the internal survey work by personnel of the SF Office, improvements were made in internal records of material-balance areas as well as in the storing of material not in use. Completion of the numerous new buildings contributed heavily toward solving many storage and handling problems.

Outstanding problems anticipated in 1953 are: (1) storage and/or decontamination preparatory to returning to production channels numerous items containing highly irradiated U^{235}, since this is becoming a major problem within ORNL; (2) preparation and issuance of a manual covering SF accountability, health and safety, and security and criticality procedures for ORNL; (3) an expected increase in shipments of fissionable material, since it is apparent that MTR fuel assemblies will continue to be fabricated at ORNL and subsequently shipped to Arco, and it is anticipated that shipment of expended LITR and BSTR assemblies will also be shipped to Arco; and (4) forthcoming new programs, particularly Thorex, ARE, Lid Tank Experiment, Tower Shielding, HRE No. 2, and possibly ADP, on which accountability procedures will be important.

[redacted]