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1954

OPERATIONS DIVISION ANNUAL REPORT

FOR YEAR ENDING DECEMBER 31, 1953

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OPERATIONS DIVISION ANNUAL REPORT

for Year Ending December 31, 1953

by

A. F. Rupp

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OPERATIONS DIVISION ANNUAL REPORT

REACTOR OPERATIONS

ORNL Graphite Reactor

The operation of the Graphite Reactor in conjunction with that of the LITR has become quite routine, and it was possible to allow the work force to be reduced gradually to three operators and one supervisor per shift. Both the Graphite Reactor and the LITR are operated with this man power, to which four operators scheduled on fixed shifts are added so that a larger work force is available during the day and evening shifts.

By combining the operation of the two reactors it has been possible to operate the LITR for only approximately $140,000 more than it would have cost to operate the Graphite Reactor alone. A summary of reactor operations is shown in Table 1.

The bonded slugs charged to the Graphite Reactor in 1952 have permitted operation at a slightly higher power and metal temperature in 1953 than in 1952. However, the increase in power has not been as great as that expected, and more slugs have ruptured in 1953 than in any previous year. Only 14 of the ruptured slugs were bonded, the remaining four were unbonded and of the type formerly used. These unbonded slugs were being irradiated for the production of radioiodine so as to conserve the bonded-slug supply.

The rate at which the unbonded slugs ruptured (only 150 to 175 were in the reactor at one time, the rows were short, and the slugs remained for only about two months) indicates that perhaps the reactor power is actually higher than that indicated by the present air-flow calibration. Also, the temperature of the slugs has been considerably higher than that experienced when the reactor contained unbonded slugs. Since the temperatures are measured by thermocouples welded to the ends of the slugs, the higher temperature appears to indicate that either the bonded slugs have better thermal contact with the jackets at the ends or that the reactor power is actually higher than that indicated. A new calibration will be made.

Most of the ruptures occurred between May 18 and October 18. After the latter date, when cool weather had set in, the reactor power was maintained at 3700 kw, which caused the maximum slug temperature to be decreased from about 280 to 250°C. This indicates that the bonded slugs, as well as the unbonded slugs, rupture rapidly if a certain temperature is exceeded. However, the evidence referred to above indicates that the reactor power is actually higher than that indicated by air flow and that the bonded slugs

TABLE 1. SUMMARY OF REACTOR OPERATIONS

	YEAR 1953		YEAR 1952	
	Graphite Reactor	LITR	Graphite Reactor	LITR
Total energy, kwhr	30,351,453	15,198,891	28,995,986	9,399,538
Average power/operating hr, kw	3,749	1,997	3,685	1,287
Average power/24-hr day, kw	3,465	1,735	3,301	1,070
Lost time, %	7.57	13.1	10.42	16.8
Slugs charged	1,165		43,521*	
Enriched fuel pieces charged		19		18**
Research samples charged	797	227	1,001	113
Radioisotope samples charged	2,468	159	2,188	81

*Graphite Reactor loaded with bonded slugs.

**Includes many lightweight fuel elements.

[redacted]

have permitted higher power operation than was possible with unbonded slugs. A summary of the slug-rupture data is given in Table 2.

At the present rate of consumption it appears that there should be enough slugs to last about eight years. However, no unusual demands for slugs have occurred in the past year, and a requirement for a few thousand slugs would materially alter this estimate.

It has been found that some of the slugs in the reactor which were not beta transformed when fabricated have been increasing in length. Several lots of slugs, for example, have been measured by the Metallurgy Division after they were discharged along with a ruptured slug and have shown increases in length to a maximum of $^3/_{16}$ in. per slug after 464 days of irradiation. In another lot of 23 slugs irradiated for 569 days, the average increase in length was 0.084 inch. These measurements have been confirmed by measuring the total length of the slug rows in the reactor. After six months, some of the rows containing these slugs which were not beta transformed have increased as much as 6 in. in length. Of eight rows measured, the length increased between 3 and 6 in. in a total length of about 14 feet. The reactor contains a considerable number of slugs which were not beta transformed because it was not learned that the beta transformation was incomplete in these slugs until June 1952; by this time, 26,000 slugs had been charged into the reactor.

The fuel-slug supply at the end of the year was as follows:

In the Reactor	
Bonded	41,991
Unbonded	118
Experimental bonded (1866)	15
Donut	21
In the Vault	
Bonded	10,263
Unbonded	620
Rejects	140
Donut	35

In June and July 1952, approximately 540 tantalum slugs were charged in the peripheral rows of the reactor. Eighty-three of these slugs were discharged in March 1953 and were checked for leakage at the Dugway Proving Ground. Of the 63 slugs opened, none were found to contain water, and it was planned to discharge the remainder about August 1, 1953 so they could be used by the Army Chemical Corps. It was later decided that the slugs could not be used, and they were discharged on August 31 and are being held in the canal until they can be transferred out for burial.

Excess reactivity was short during a large part of 1953, but after discharge of the tantalum, the excess was sometimes more than 150 inhours. To reduce the excess reactivity, several channels were left empty as they were discharged. Also, a number of slugs containing gold were charged in the empty rows so that Hg^{198} would be produced.

Stored-energy measurements on samples of graphite removed from one of the shim-rod channels indicated that about 15 cal/g was released up to 200°C. The total stored energy was thought to be about four times this amount.

The operating galvanometer neutron chambers have been replaced with flow-type compensated chambers similar to those used at the LITR. The safety chambers will also be replaced with flow-type uncompensated chambers. This will simplify maintenance and supply of spare chambers, since the LITR and the Graphite Reactor will use the same types of chambers.

No changes occurred in the mattress plates during the year. Because of the small number of slugs discharged, the wear on the plates was not as great as it was last year when 42,128 slugs were discharged. The north plate was last changed on November 24, 1952, and the south plate was changed on December 1, 1952. Different channels (those above the south plate) were used for radioiodine production so that no discharges ordinarily will occur on the north mattress plate. This should make it possible to use the north plate for a longer time, while the south plate will be changed as needed.

The canal continued to be quite contaminated, mostly because of Co^{60} dissolved from cobalt being stored in the canal. The activity in the canal water generally varied between 1000 to 2000 counts/min/ml when measured on a scintillation counter. The activity in the section where cobalt was stored (separated from the rest of the canal by a wooden dam) generally was between 5,000

TABLE 2. SLUG RUPTURES DURING 1953

RUPTURE NUMBER	CHANNEL NUMBER	DATE DISCOVERED	DAYS IN REACTOR	APPROXIMATE TEMPERATURE (°C)	POSITION*	LOT NUMBER	BETA TRANSFORMATION (%)	REMARKS
				Bonded				
109	1163	1/5	56	220	21	6213	100	
111	2071	3/30	363	265	14	114	25	
113	2070	5/25	419	240	15	110	0	
114	2178	6/1	385	175	33	145	100	
115	1272	6/15	399	95	19	154	75	Thermocouple slug
116	1270	6/22	420	160	18	138	100	
117	1777	6/22	427	175	8	135	100	
118	1872	7/9	464	270	13	113	0	Thermocouple row, found by increasing temperature of thermocouple
119	1362	7/20	445	210	9	129	100	
120	0968	7/20	245	200	36	6213	100	
122	1872	8/12	30	207	3	6137	100	
123	1775	8/17	476	242	20	138	100	
124	1559	9/14	490	165	17	149	100	
125	1670	10/12	559	157	23	110	0	
				Unbonded				
108	1969	1/1	80	240	1			Iodine row
110	1964	1/19	98	240	1			Iodine row
112	1964	5/18	55	240	2			Iodine row
121	1964	8/3	84	230	1			Iodine row

*Determined by counting from west end of row.

to 20,000 counts/min/ml. The effluent water from the canal was discharged through ion-exchange columns to remove radioactive cations. This reduced the radioactivity of the effluent water to between 30 and 100 counts/min/ml, but the pH was so low (due to replacement of cations by H^+) that the water could not be returned to the canal. Instead, the effluent water was discharged to the settling basin at a rate of about 20 gpm. It may be possible in the future to add anion columns so that the deionized water can be returned to the canal and recirculated. It is hoped that all the cobalt will be removed from the canal in 1954, and when this is done it should be possible to keep the canal water much cleaner.

Both fans operated normally during 1953. Bearing inspections were carried out at intervals on both fans.

The filter house operated satisfactorily; the glass-wool filters in cell No. 2 were changed on April 9, 1953. At the current rate of change of pressure drop across the house, it is expected that the glass-wool filter in cell No. 4 will be changed in the spring of 1954.

Low-Intensity Test Reactor

The major event of the year in the operation of the LITR was the doubling of the power on September 2, 1953. Since that date, the reactor has operated routinely at 3000 kw with no indication of difficulty. The increase in power was made possible by adding an extra inch of lead shielding around the exit-water line, by adding an extra course of barytes concrete blocks around the seal tank, and by increasing the shielding in a number of other places as required. The capacity of the pumping system was increased by enlarging the inlet lines to the pumps, and heat removal by the forced-air fluid coolers was supplemented with the shell and tube heat exchanger whenever the outside temperature was above 80°F.

Water-drop tests were made at 1500, 1900, and 2300 kw for testing the efficacy of the spray system in cooling the fuel elements, following loss of water from the reactor tank. The system operated very satisfactorily, and temperatures indicated by thermocouples inserted between the fuel plates did not rise above about 92°C.

The major limiting factor in the life expectancy of the LITR is now believed to be the condition of the neoprene gaskets between some of the reactor tanks. Test samples of neoprene located similarly to the gaskets are being irradiated to determine the approximate damage rate.

A bypass demineralizer installed on January 14, 1953 has proved to be very successful in maintaining the circulating water at a high purity and in reducing the corrosion rate of the components of the system. Prior to installation of the demineralizer, the corrosion rate on the No. 1030 steel coupons installed in the upper portion of the reactor tank was 15.3 mils/year; after installation, the rate dropped to 4.0 mils/year. The demineralizer has been operated generally as a mixed-bed unit, but operation as two separate columns – one anion and one cation – was tried and found to be inferior to the monobed type of operation. The columns have proved to be entirely too small for the system and must be regenerated every two or three weeks. It is planned to install a larger column, which will not require such frequent regeneration and which can be regenerated without removing the resins.

The turbulators (spirals inside the water tubes designed to give more turbulence and thus increase the heat transfer) were removed from the forced-air fluid coolers in April after it was found that chattering of these parts wore holes in some of the tubes. Very little loss of capacity was noticed after the turbulators were removed, and the coolers have given good service since that time. The pressure drop across the coolers is also considerably less since the turbulators were removed.

Contamination in the circulating water at the LITR has increased to approximately 300,000 counts/min/ml following the increase in power in September. An analysis of the radioactivity in the water in April showed about 58% Np^{239}, 27% Na^{24}, and 3% Mn^{56}. The larger ion column, referred to above, should help reduce the radioactivity and lessen the contamination of tools which must be put into the reactor tank during shutdown operations.

A number of corrosion samples were exposed in the reactor tank for obtaining information on the corrosion rate of reactor components. The results shown below are the highest corrosion rates found for different materials, each of which was exposed in contact with aluminum, with stainless steel, and insulated from contact with other metals.

Material	Exposure (days)	Corrosion Rate (mils/year)
Type 1030 steel	182	4.0
Aluminum (2S)	182	0.2
Beryllium	183	1.2

Further tests are being made for determining corrosion rates of these same materials in the lattice, where they receive a large amount of irradiation. Preliminary results on beryllium indicate that its corrosion rate has changed very little. The iron sample irradiated at this location is so radioactive that it will have to be defilmed and weighed in a hot cell.

A plug in facility HB-2 stuck on December 8, and considerable difficulty was encountered in removing it. To lessen the danger of plugs becoming stuck as a result of the galling on the soft aluminum walls of the holes, stainless steel liners are being fabricated for the beam holes, and plugs for the 7- and 6-in. sections are being made smaller.

As a result of better scheduling operations, it has been generally necessary to remove the top plug only once every two weeks instead of once every week, as was originally done. This materially lessens the shutdown work and the down time.

RADIOISOTOPE PRODUCTION AND SALES

The steady increase in the production and the shipment of the three major processed radioisotopes – I^{131} (+5.2%), P^{32} (+15.1%), and C^{14} (+2.3%) – was maintained throughout the year. The significant production data for these isotopes are listed in Tables 3, 4, and 5; special preparations are listed in Table 6.

The yield of I^{131} per pound of uranium processed dropped appreciably this year. A part of the

TABLE 3. IODINE-131 PRODUCTION DATA

YEAR	NUMBER OF SLUGS USED			URANIUM PROCESSED (lb)	PRODUCT SHIPPED (mc)	MILLICURIES SHIPPED PER POUND OF URANIUM USED	OPERATING COST PER MILLICURIE SHIPPED
	ORNL	Hanford 4-in.	Hanford 8-in.				
1953	828	0	4	2159	504,455	234	$0.316
1952	644	10	2	1696	488,231	288	0.329
1951	651	5	0	1693	467,544	276	0.372
1950	396	12	0	1066	310,698	291	0.627

TABLE 4. PHOSPHORUS-32 PRODUCTION DATA

YEAR	SULFUR PROCESSED (kg)	PRODUCT SHIPPED (mc)	MILLICURIES SHIPPED PER KILOGRAM OF SULFUR USED	OPERATING COST PER MILLICURIE SHIPPED
1953	555	137,100	247	$0.942
1952	463	118,668	256	1.436
1951	406	107,187	264	1.825
1950	452	95,954	212	1.990

TABLE 5. CARBON-14 PRODUCTION DATA

Total product, mc	8,091.9
Weight of $BaCO_3$, g	121.6
Average C^{14} abundance, %	19.9
Maximum C^{14} abundance, %	29.0
Minimum C^{14} abundance, %	10.0
Be_3N_2 slugs used	109
Be_3N_2 weight, g	10,890
Cost per millicurie	$3.37

TABLE 6. SPECIAL PREPARATIONS

	NUMBER	TOTAL ACTIVITY
A^{37} ampoules	21	74.5 mc
Au^{198} source	1	80.0 curies
Ba^{140} sources	2	100.0 mc
Cs^{134} sources	2000	20.0 curies
Cs^{137} sources	15	7.27 curies
Co^{60} sources	1323	5357.75 curies
Fe^{55} x-ray source	1	3.0 mc
He^3 ampoules, ~95%	18	961.0 cc
He^3 cylinders, ~5%	3	2300.0 cc
H^3 ampoules	49	49.94 curies
He^3-Zr targets	31	42.74 curies
Sr^{90} sources	14	212.0 mc

decrease can be attributed to breakdowns in the equipment, but many poor runs remained unexplained; however, the gross cost of production was 4% below that of last year. This decrease was due mainly to the 3.3% increase in production and sales without a corresponding increase in man power and materials.

The 3.5% decrease in the P^{32} yield resulted mainly because it was frequently necessary to reprocess the product before shipment could be made; the material failed to pass the precipitation test when the pH was adjusted to 7. This condition was alleviated in the latter part of the year, and yields became higher than normal after several minor changes were made in the operating techniques and the chemistry of the process, most significant of which probably was the use of a purer grade of sulfur for target material.

Almost half of the 34.4% decrease in the cost of production of P^{32} was due to the increase in the number of shipments made without increasing man power and material. A large portion of the balance may be attributed to decontamination and re-use of sulfur irradiation cans (the cans are now being re-used six times – formerly only one irradiation per can was obtained) and the re-use of the glass extractor tubes which was made possible by designing a new type of tube which permits the removal of the "hot" waste sulfur.

No difficulties were encountered in the production of C^{14}. The C^{14} isotopic abundance of the product was the highest yet attained. A storage garden, consisting of six 12-in.-dia stainless-steel-lined underground holes which can be sealed and kept dry, was built for storage of the 955 Be_3N_2 slugs on hand. These slugs were formerly stored in the reactor-building canal. The production cost listed in Table 5 does not include the cost of procuring or irradiating the Be_3N_2.

The production and shipments of all radioisotope products are shown in Table 7. It will be noted that the fission products, Cs^{137} and Sr^{90}, are steadily gaining in sales. When Co^{60} shipments are subtracted, the total amount of activity shipped increases to 50.5%.

Improvements in production and increased sales volume permitted reductions in the sales prices of several important radioisotopes, as follows: Pm^{147}, $20/mc to $0.50/mc; Tl^{204}, $5/mc to $0.75/mc; Cs^{137}, $0.50/mc to 2.5¢/mc.

Data on radioisotope sales and income are given in Table 8. The number of shipments continued to climb (+12.6%), as did income (+15%), despite a $147,378 reduction in AEC cancer subsidy money.

A significant increase is shown in the Technical Cooperation Program income (+350%) because of shipments to the Canadian Radioisotope Production Group to furnish radioisotopes for its customers while the Chalk River Reactor was being repaired.

RADIOISOTOPE DEVELOPMENT

New Iodine-131 Unit

The new I^{131} production unit was practically completed during the year. Contractor work was finished in March, but delays in obtaining equipment and craft man power extended the time required for equipment installation and instrumentation.

TABLE 7. RADIOISOTOPE PRODUCTION AND SALES

	AMOUNT PRODUCED DURING 1953 (mc)	AMOUNT SHIPPED (mc)	
		1953	1952
Antimony-124	2,585	149	236
Antimony-125	102	6	2
Argon-37	75	54	0
Arsenic-73,74 (86-in. cyclotron)	46	3.5	0
Arsenic-73,74 (other cyclotrons)	0	0	6
Barium-140	4,583	689	928
Beryllium-7	509	83	106
Cadmium-115	446	26	17
Calcium-45	6,291	2,039	1,378
Carbon-14	8,092	5,896	5,763
Carbon-14 organics	0	46.29	100.05
Cerium-141	159	132	414
Cerium-144	975	401	383
Cesium-134	2,712	596	542
Cesium-137	83,136	20,693	5,322
Chlorine-36	81	4.8	1.4
Chromium-51	13,328	3,939	1,641
Cobalt-56	3,000	3,000	0
Cobalt-57,58	492	4	1
Cobalt-60 (metal)		6,181,000	9,633,000
Cobalt-60 (solution)	0	801	875
Fission products (mixed)	8,089	333	3,352
Hydrogen-3		30,918	210,647
Indium-114	682	23	0
Iodine-131		514,000	489,000
Iridium-192	10,300	192	0
Iridium-192 (metal)	12,000	12,000	0
Iron-55	162	89	142
Iron-55,59	267	61	139
Iron-59	410	248	200
Manganese-52,54 (other cyclotrons)	425	21	20
Mercury-203	15,273	367	579
Neodymium-147	13	1	5
Nickel-63	297	35	78
Niobium-95	682	141	194

TABLE 7. (continued)

	AMOUNT PRODUCED DURING 1953 (mc)	AMOUNT SHIPPED (mc)	
		1953	1952
Phosphorus-32		137,000	119,000
Polonium-210		244,365	67,653
Polonium-210–beryllium		378,386	0
Praseodymium-143	60	8	3
Promethium-147	0	417	38
Ruthenium-103	843	333	51
Ruthenium-106	42,852	9,496	547
Scandium-46	13,482	180	120
Selenium-75	12,018	451	588
Silver-110	720	349	272
Sodium-22 (other cyclotrons)	137	103	32
Sodium-24	41,681	360	0
Strontium-85	0	1	7
Strontium-89	1,938	1,513	10,507
Strontium-90	91,858	61,375	20,268
Sulfur-35	59,278	7,527	8,056
Tantalum-182	384	402	0
Technetium-99		0.049	0.10
Thallium-204	0	1,247	464
Tin-113	61	31	22
Tungsten-185	935	115	57
Yttrium-91	1,337	571	2,041
Zinc-65 (86-in. cyclotron)	14	3	4
Zirconium-95–niobium-95	11,302	2,193	1,828
Total	472,453	7,617,727	10,586,784
Total minus Co^{60} metal	472,453	1,436,727	953,784

	NUMBER SHIPPED 1953	NUMBER SHIPPED 1952
Service irradiations (reactor)	609	471
Service irradiations (other cyclotrons)	52	55
Unprocessed units	1,687	2,491

The unit is scheduled for completion by February 1954 (Fig. 1).

TABLE 8. RADIOISOTOPE SALES AND INCOME

	YEAR 1953	YEAR 1952
Total number of shipments	12,036	10,691
Total shipping weight, lb	305,520	324,812
Income (total)	$1,333,057	$1,159,233
Foreign	$51,015	$16,760
Project	$87,569	$143,918
Domestic	$862,751	$572,552
Cancer*	$229,078	$376,456
Technical Cooperation Program	$10,208	$2,228
Sales to Carbide (Oak Ridge)	$90,681	$43,584
Civilian defense	$1,755	$3,735
Production cost (total)	$1,122,423	$989,101

*Income subsidized by AEC; does not include cash received.

Iodine-131 Purification

A new I^{131} purification process, the deposition of carrier-free iodine on silver metal surfaces, was investigated. Satisfactory results were obtained except in the presence of large amounts of nitrate. By the end of the year, the new process appeared to have some chance of replacing the distillation purification now used.

Sulfur Target Material for Phosphorus-32

The P^{32} process required virtually no attention from the Development Group this year. It was demonstrated that Al_2O_3 could be substituted for MgO for sulfur target material purification; however, a new commercial supply of sulfur was found to be satisfactory without further purification.

Separation of Fission Products from Waste

The fission-product separation process (FPP process) was a major radioisotope development problem during the year. The objective of this process is to use tank-farm wastes, principally Purex 1AW, for the production of large quantities of the most important long-lived radioisotopes: Cs^{137}, Sr^{90}, Ce^{144}, Ru^{106}, Pm^{147}, and Eu^{155}. Several types of equipment were used with various flow sheets in the 100 curie/week unit located in the south tank farm; the feed was Purex 1AW-2AW from the Scrup program. The principal difficulty encountered was the behavior of large amounts of iron (in the feed material) in the various precipitation steps which caused consequent difficulties with filtration. A method of precipitating most of the iron by the hydrolysis of urea was developed and used on a large scale to selectively precipitate $Fe(OH)_3$ in a form which would not carry appreciable amounts of fission products other than ruthenium. Another finding of interest was the failure of the cation-exchange column as a result of radiation damage during the purification of Cs^{137} batches as small as 50 curies. The removal of this ion-exchange step and its replacement with the precipitation of $Al(OH)_3$ by slow addition of ammonia gas marked the end of trials of ion-exchange resin for process steps in which radiation damage is possible. Stainless steel vessels were found to be completely unsuitable for the precipitation of ceric iodate because of surface-reducing effects. A process for extracting extraneous iron from fission-product solutions in 10 *N* HNO_3 with TBP was also explored and found to be worthy of further study as an alternate process. Approval of the proposed Multicurie Fission-Product Plant was not obtained and no further process equipment or plant layout design was attempted.

Kilocurie Cesium-137 Source

Methods of purifying Cs^{137} and fabricating sources suitable for teletherapy machines were tried. A variation of the alum process was found to be quite satisfactory for the purification of cesium, as well as for the primary separation. Sulfate ion was successfully removed by passage of the material through an anion column at a rate high enough to prevent radiation damage to the resin. Experiments showed that, while Cs_2SO_4 would be a desirable compound for a compressed source, the work involved in drying the material and converting from $CsHSO_4$ was prohibitive. Cesium chloride was tested and found to be satisfactory, even though it is more volatile than Cs_2SO_4. It was demonstrated that $Cs^{137}Cl$ can be pelletized to a density of 3.37 g/cc, with a resultant specific activity of 20 curies per gram of

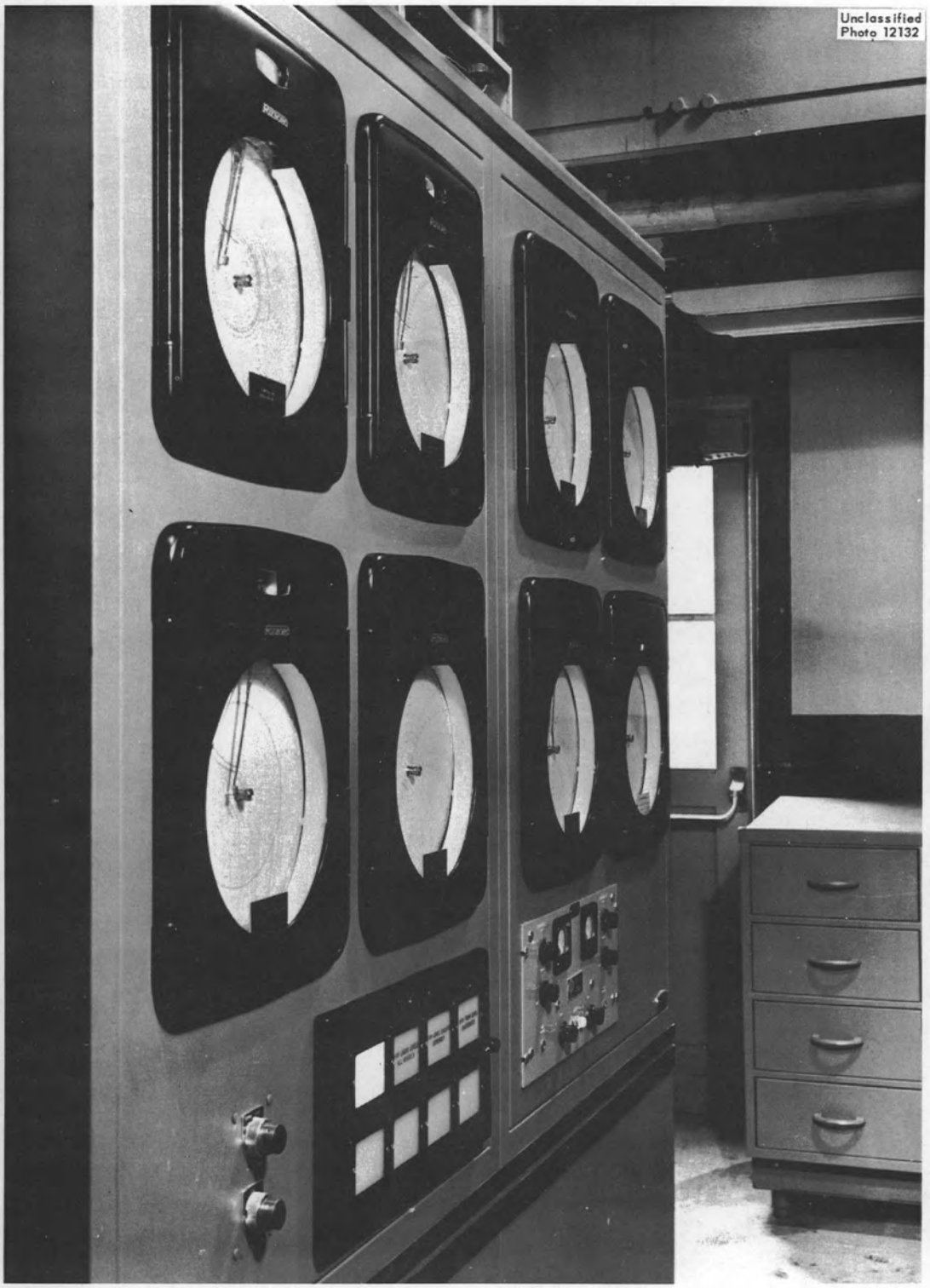

Fig. 1. View of New I^{131} Production Unit Panel Board, December 1953.

gross weight. A 60-curie test pellet was made and photographed by its own light (Fig. 2).

While it was originally intended to do the fabrication of the 1500-curie source in revised equipment in the old Ra-Be cell, it was shown that this work could be done best in the remote manipulator cell (Argonne hands). This demonstrated clearly that future emphasis should be put on manipulator cells for this type of work.

Studies on the Production of Europium-152,154

Many workers have been interested in the possibilities of using $Eu^{152,154}$ as a substitute for Co^{60}, particularly for teletherapy machines, since the cross section for production is high and the half life was thought to be about 5 years. It has been recently found that the half lives are 12 to 16 years, and it was shown by the Development Group that the burnup of the $Eu^{152,154}$, combined with the increased half life and relatively low gamma output, made this source less attractive. A complete production curve was calculated from available data, and two test pellets of compressed Eu^{203} (99.2% pure), 9.5 mm in diameter and 1 mm thick, accompanied by cobalt monitors are being irradiated in the LITR. This is a long-term test to see if the maximum predicted on the curve (27 curies/g) and the production rate are correct.

Fig. 2. Cs^{137} Cl Test Pellet, Photographed by Its Own Light.

After 169 days of irradiation, the observed activity was slightly below the calculated values. This experiment will continue through 1954.

Separation of Ruthenium-103,106

The standard method of $KMnO_4$ oxidation of ruthenium to RuO_4 with subsequent distillation of the latter was modified to allow direct distillation from UNH dissolver solution, thereby allowing a fast separation, which is particularly useful for producing the relatively short-lived Ru^{103}. Ruthenium-106 was found to accumulate on $Fe(OH)_3$ residues during the urea hydrolysis in the FPP process; the standard $KMnO_4$ process was also adapted for this feed, with over-all yields of about 65%.

Promethium-147 Sources

Two experimental 10-curie Pm^{147} sources were made by incorporating $Pm_2^{147}O_3$ into a glass which was fused to platinum disks at 1100°C. The sources were completely free from smear contamination and were resistant to severely corrosive reagents, such as boiling nitric acid. These sources are being evaluated by an aeronautical engineering group for measuring shock waves by the variations in beta-particle penetration of air at different densities.

Separation of Promethium-147 from Americium-241

Promethium-147 and Am^{241} have almost identical chemical properties, although the former is a rare earth of the lanthanide series and the latter is a member of the actinide series. Tests were made on the ion-exchange fractionation of these elements with concentrated HCl. By using 13 *N* HCl, produced by absorption of HCl gas in 37% HCl solution, decontamination factors for the elimination of Am^{241} from Pm^{147} of about 50 were obtained. There will be an opportunity to try this method on batches as large as 1000 curies of Pm^{147} during the coming year.

Purification of Mixed FPP Rare Earths

Rare-earth fractions from the FPP process are contaminated with Fe, Cr, Ni, Ca, and other minor constituents. Two methods were used for purification: (1) extraction of the rare earths into TBP from 16 *N* HNO_3, followed by selective stripping at various HNO_3 concentrations; and (2) precipitation of the rare earths, sometimes by using lanthanum carrier, as oxalates from acid solution. The better results were obtained with the oxalate method, which gave a yield of 90% with simple equipment and operations. However, the TBP method may be used also for fast separations on short-lived rare earths when yield is not so important.

Carbon-14

The development of specifications for Be_3N_2 target material and the negotiation to obtain the proper material in pellet form were the main jobs done on C^{14} during the year. The Brush Beryllium Company will start delivery next spring on 400 lb of pelleted Be_3N_2 containing 200 to 300 ppm of carbon (carbon content of present material is 1200 ppm). This material will be irradiated at Arco and at Hanford for at least one year; the Laboratory should be able to produce radiocarbon with an isotopic abundance of more than 50% C^{14} from these targets.

Small amounts of $HC^{14}N$ and $C^{14}H_4$ were separated directly from the Be_3N_2 process gases by adsorption on silica gel and by chemical absorption methods. Analyses are not complete, but it is apparent that $C^{14}H_4$ is the major C^{14} constituent of the process gases. The $HC^{14}N$ was precipitated as $AgC^{14}N$ and noted (by approximation methods) to be of very high isotopic abundance.

Strontium-90

Improvements were made in the old HCl-ether method of separating strontium from barium by precipitating the latter as $BaCl_2$ in very concentrated HCl solution instead of in the HCl-ether mixture. A technique for easily generating 10 to 13 *N* HCl by absorbing HCl gas in 38% HCl was worked out, and solubility curves for $BaCl_2$ in various concentrations of HCl at several temperatures were made.

A mercury-cathode electrolytic cell was used to reduce the heavy-metal contamination of product solutions to less than 10 ppm, which is required for Sr^{90} to be used for activating phosphors.

Hydrogen-2 or Hydrogen-3–Zirconium Targets

Work continued on the preparation of very thin evaporated films of zirconium containing isotopic hydrogen. The procedure developed was successfully used for routine production of targets during the year. Data were collected to correlate the bremsstrahlung radiation from the targets with the

actual H^3 content; this work will continue during 1954.

A method was developed to bond zirconium foil to the backing metal for the production of "thick" zirconium targets. The bonding is accomplished by coating the backing material with an evaporated film of iron, onto which the zirconium foil is lightly pressed and then heated by induction at 1100°C; a metallurgical bond is obtained. Very satisfactory targets were made in this manner, and the method may be adaptable for covering other metals with a thin zirconium cladding.

Helium-3

A system for purifying and packaging He^3 was built and put into routine operation. Helium-3 that contained as little as 0.01 ppb of H^3 was produced.

Krypton-85

Fission-product gases were received from Arco, and a portion of these gases was processed to produce pure Kr^{85} and stable fission-product xenon. A new shielded vacuum system is being designed, and this equipment will be used to process the remainder of the gas because of the hazard from gamma radiation when 100-curie batches of Kr^{85} are handled. The molecular weight of the fission-product xenon was measured and found to be 131.4, which is reasonably close to the value calculated from fission-product yields.

Cyclotron-Target Processing Methods

The major portion of the target-processing work was the chemical processing of targets from the ORNL 86-in. cyclotron, both for the production of radioisotopes and to determine cyclotron yields. The ORNL machine was found to be very suitable for producing Co^{56}, Co^{57}, $Mn^{52,54}$, Be^7, Zn^{65}, $As^{73,74}$, Y^{88}, and Bi^{207}. Chemical and handling methods for processing the very large, cumbersome targets from this machine were developed. Low-specific-activity Na^{22} made by irradiating normal sodium with protons was produced in fair yield, and consideration is being given to marketing this material if there is a demand for it.

Iridium-192

Iridium metal or its oxide is extremely difficult to dissolve; however, Ir^{192} solution is needed for dispensing small amounts for the radioisotope program. A method was used successfully whereby highly radioactive iridium pellets were chlorinated with chlorine gas in molten NaCl at 700°C. A water solution of the melt contained sodium chloriridate. It was noted that the intense radiation tends to cause reduction of the iridium to a lower valence state when the solution is allowed to stand.

The gamma radiation output of Ir^{192} was determined by measuring carefully assayed solutions, dried down to point sources, with a standardized gamma chamber; the measurements were made (in air) 10 ft above the ground to minimize scattering. The value found was: 1 curie of Ir^{192} = 1.82 r/hr at 1 meter.

RADIOACTIVE-WASTE DISPOSAL

The total combined activity discharged to White Oak Creek from the settling basin and the retention pond during 1953 was 429 curies. This discharge was 86% of that in 1952 and 250% of that in 1951, which was the lowest on record. (See Table 9 for other comparisons.)

The largest single source, which contributed more than 30% of the total, was a leaking group of valves in the north tank farm which served to control the flows of waste from the hot pilot plant. This leak, which began late in 1952, was not

TABLE 9. WASTE DISCHARGED TO WHITE OAK CREEK

YEAR	VOLUME (gal)	BETA CURIES FROM SETTLING BASIN	BETA CURIES FROM RETENTION POND	TOTAL ACTIVITY (beta curies)
1953	239,356,000	289	140	429
1952	268,176,000	411	87	498
1951	297,590,000	169	3	172
1950	266,350,000	172	15	187

located until March of this year. It was found, after extensive excavation through the north tank farm, to be channelling through the ground and entering the retention-pond lines through loose joints in the pipe. The entire valve group has been replaced with a stainless-steel-lined, concrete diversion box, but it has not yet been tied into the system.

The second largest known single source of activity was the valve pit on the west side of Building 3026 through which the Rala wastes pass. About 35 curies of activity was discharged to the settling basin in November when two leaks developed, one in the chemical-waste line and the other in the metal-waste line.

The waste evaporator contributed approximately 19 curies during the entire year, and an accidental spill of cesium in the Radioisotope Area, Building 3029, contributed 10 curies. The balance of approximately 230 curies came from normal operations and some minor spills, the activity of which could not be estimated.

As in previous years since the waste evaporator was put into operation to handle high-activity wastes, the large volume of low-activity process wastes and accidental discharges which could not be processed through the waste evaporator accounted for 96% of the total activity put into White Oak Creek. A study, begun in 1952 and completed early this year, showed that most of the activity could be removed by processing the combined, diluted wastes through a commercial type of water-treatment plant. The construction of this plant is now awaiting the approval of the AEC.

The control of the wastes entering the process-waste system was considerably improved this year with the addition of monitors which measure the total volume of the various streams in the system and sample them. The only main stream not monitored is the one from the Research Building (4500); the equipment installation could not be completed because of lack of Mechanical Department man power.

A serious effort was made this year to reduce the volume of water entering the process-waste system, since it is anticipated that the proposed water-treatment plant will be installed. The flow into the system was reduced by 11% by diverting the cooling water from air conditioners and other equipment from the process-waste system to the storm sewers. An even greater reduction is expected as soon as some long-standing work orders for diverting several large unnecessary flows into the system from the Radioisotope Area, the Metallurgy Building 2000, the Health Physics Building 2001, and the Physics Building 2004 are processed.

The waste evaporator operated satisfactorily throughout most of the year. Its capacity remained adequate, with an average of 258 gph being received for processing; this rate was essentially the same as that in 1952. Evaporator operation is shown in Table 10.

Approximately 227,000 gal of concentrated radioactive waste containing 7716 curies of beta activity was transferred from the tank farm to the chemical-waste storage pit No. 2. The total transferred to the pit thus far is 270,000 gal containing 8669 curies (Fig. 3).

A speed-up in the transfer of waste to the pit and an appreciable reduction in exposure of man power to radiation was accomplished by using an old, salvaged gasoline trailer which was excessed by the AEC and converted for waste-hauling purposes at the Laboratory. This equipment was far superior to

TABLE 10. WASTE-EVAPORATOR OPERATION

YEAR	WASTE PROCESSED (gal)	AVERAGE PROCESSING RATE (gal/hr)	BETA CURIES IN FEED	BETA CURIES DISCHARGED TO SETTLING BASIN	DECONTAMINATION FACTOR
1953	2,259,516	258	147,790	19.45	7602
1952	2,258,155	257	217,474	23.77	9149
1951	2,474,254	282	57,285	7.24	7912
1950	2,481,207	283	22,631	7.56	2994

Fig. 3. View of Storage Pit No. 2 Showing Transfer Tank Truck.

the 500-gal Dempster Dumpster tanks originally used.

Construction of a pipe line connecting the tank farm to the waste-storage pit is expected to be completed early in 1954. This project will eliminate the hazardous hauling of waste by truck, will probably eliminate the need for concentrating wastes by the waste evaporator, and will greatly reduce the amount of effort and money expended in the disposal of liquid wastes.

The metal-recovery program was started by the Chemical Technology Division in January and continued throughout the year. Approximately half the total uranium stored in the tank farm has been removed during the operation of this project. The metal-waste tanks, which have a combined capacity of 583,000 gal, were approximately half empty at the end of the year; the available operating free space was the greatest in many years.

The over-all Laboratory liquid-waste disposal picture appears brighter at present than it has for many years (see Table 11). With the addition of the monitoring tanks and the telemetering system to the "hot" waste system and the monitors to the process-waste system, a better control of the wastes is now possible than ever before. The construction of the pipe line to the waste-storage pit will eliminate the need for more chemical-waste storage tanks and will provide a safer and cheaper way of permanently disposing of the "hot" chemical wastes. The construction of a water-treatment plant for handling the large-volume process wastes, if approved, will eliminate most of the activity now being discharged to White Oak Creek.

TABLE 11. WASTE-DISPOSAL OPERATING COST

1953	$199,704
1952	208,331
1951	288,465
1950	274,119

MISCELLANEOUS OPERATIONS

Water Demineralization

Multiport valves gave trouble occasionally and had to be repaired to prevent regeneration-solution leaks. Also, the No. 1 unit was found to have a shortage of several cubic feet of anion resin. After the resin was replaced, the unit operated very satisfactorily. It now appears that the No. 2 unit also lacks some of the anion resin. The deaeration equipment and a considerable part of other unused apparatus in the water-treatment plant will be salvaged as rapidly as possible.

The amount of water demineralized in 1953, compared with that in 1952, is as follows:

Water Demineralized

Year	Gallons
1953	4,467,118
1952	4,747,664

Liquid Hydrogen

An explosion occurred on January 23 when a carpenter drove a nail through a high-pressure hydrogen line. Following this accident, the liquid-hydrogen production unit was relocated in a small building on the east side of Building 3001 where it is now functioning satisfactorily.

Cyclotron Radioisotopes

Eighty-four targets were scheduled to be irradiated in the ORNL 86-in. cyclotron, five of which required chemical processing.

Fourteen targets were received from university cyclotrons for processing, as follows: Washington University, 1; University of California, 6; University of Pittsburgh, 6; Massachusetts Institute of Technology, 1.

Reactor Irradiations at Other Sites

The irradiation of target materials at Hanford continued to be an extremely important part of the radioisotope program. Receipts of material were less this year (226 cans) than last (422 cans), but this was largely due to the heavy discharge of Be_3N_2 for C^{14} last year, most of which remains in storage.

Irradiations were started in the MTR, particularly to produce high-specific-activity Co^{60}, but only two cans of Co^{60} and a few test cans of other target materials were received by the end of the year.

Activation Analyses

Twenty-nine requests were received for activation analyses, and 27 analyses were performed (by the Analytical Chemistry Division). The total income was $11,009.00 and costs were $12,437.00, giving a loss on this activity of $1,428.00. A slightly greater volume of business would allow this program to operate on a break-even basis. Some support is being given by the radioisotope program in the development of radioautographic methods which are applicable to commercial work. If successful, this service will also be offered to the public.

RALA

Approximately 68,000 curies of product was shipped to Los Alamos this year in two double shipments from two runs. The first run yielded approximately 17,000 curies, and the second run yielded 51,000 curies. A third run, and the only other one requested by Los Alamos, yielded no product as the result of equipment failure and processing difficulties.

The quantity of product from the first run was acceptable but was at least 13,000 curies short of the 30,000 curies requested by the users. One of the original process filters, which had been consistently causing difficulty by plugging before this run, was replaced by one with a much larger filter area. Although the filter worked very satisfactorily, it was found that the precipitated product could not be completely removed from the filter because of the shape of the filter media and the size of the connecting pipes, both of which were limited by the geometry and the size of the filter cubicle.

The second run was quite successful. The only major difficulty experienced was the jamming of the slug chute on two occasions.

No shipment was made from the third run. The basic reason for all the difficulties encountered during this run was the plugging of the process filters. A complete filtration of a batch was impossible in almost every case, and it became necessary to resort to many changes in the operating procedures. Because of frequent unplugging operations on the filters, by using air and water backwashes under pressure, serious leaks developed in the filter-cubicle equipment and caused very high losses of product. The losses through the leaks, plus the excessive decay losses, that were incurred during the three weeks the run was in progress left too little product for shipment.

The quality of the product shipped this year was, as last year, not completely satisfactory. Los Alamos was forced to perform special operations on each batch to remove organic "crud" (which is probably residue from the ion-exchange column) before the material could be used.

Most of the effort expended on the Rala project in the last several years has been in decontaminating, repairing, and testing the equipment, which has become obsolete and is being used far above its original design capacity. This undesirable situation, which was reviewed in last year's annual summary,[1] was further aggravated in 1953. The amount of time available between repairs and tests was scarcely more than adequate for the three runs made. It would have been impossible to make the six runs originally scheduled if Los Alamos had requested them.

The status of the Laboratory's proposal to build better permanent Rala production facilities has been under consideration for over two years and is still unsettled.

SF MATERIAL CONTROL

One of the major accomplishments of the SF program during 1953 was the compilation and publication of the new *SF Procedures Manual for Oak Ridge National Laboratory X-10 Area;* measurement methods, which are not included, will be issued in the early part of 1954. The manual, as issued, covers procedures for requesting and handling SF material, health and safety, security, and nuclear safety, as well as SF accountability procedures.

During the year, 124 requests for material were received from personnel within the Laboratory. Fifty-five requests were received by the Laboratory to provide material for other installations. All material requested by Laboratory personnel was delivered or scheduled for delivery; the major items furnished are listed in Table 12. The material requested for other installations was shipped or scheduled for shipment; Table 13 is a list of the shipments made. A comparative summary of the SF material activity for 1952 and 1953 is given in Table 14.

During the year, 302 shipments were received by the SF Office, including 7 carload lots and 9 trailer-load lots; 415 shipments were made, including 1 carload lot and 41 trailer-load lots.

In the interest of simplifying SF accountability for personnel holding small quantities of material, the SF Accountability Office encouraged those who were qualified by virtue of the quantity of material held to change their accountability from a status based on routine procedure to one based on research issuance. The new SF manual contains the Laboratory's procedure for requesting a research

[1] See *Operations Division Monthly Report for Month Ending December 31, 1952*, ORNL-1485, p. 22.

issuance. This provision was also brought to the attention of research and supervisory personnel by means of an official bulletin and by personal contact. While very few people took advantage of this provision in 1953, it is expected that all who are eligible will do so during 1954.

Clarification of information relative to SF accountability was received from the AEC. AEC Staff Paper 213/71 proposed that SF materials be accounted for in a manner commensurate with their economic value. This philosophy parallels the one ORNL has followed. While the general SF

TABLE 12. MATERIAL DELIVERED TO LABORATORY PERSONNEL

MATERIAL	QUANTITY	USE
Normal uranium metal	2500 lb	Lid tank experiment
	1000 lb	Metallurgical experiments
Beryllium metal chips	1400 lb	Shielding experiments
Depleted uranium slugs	3850 kg	Metal recovery for Brookhaven National Laboratory
Depleted uranium waste	6636 kg	Metal recovery for Harshaw Chemical Co.
Enriched uranium metal	3.5 kg	Fabrication of modified MTR assemblies for Argonne National Laboratory
	5.1 kg	Fabrication of TSF assemblies
	64.4 kg	Fabrication of MTR assemblies
	3.1 kg	Fabrication of LITR assemblies
	10.8 kg	Rolling foil for Y-12
Plutonium nitrate	0.14 kg	High g/ton plutonium isotope separation
Irradiated thorium metal	1143 slugs	Thorex program
Irradiated uranium metal	474 slugs	Rala process

TABLE 13. MATERIAL SHIPPED TO OTHER INSTALLATIONS

MATERIAL	NUMBER	QUANTITY (kg)	FOR
U^{235}-Al fuel assemblies	282	49.2	MTR fuel
	30	4.7	Convair reactor fuel
	50	8.27	Argonne National Laboratory
U^{235}-Al control rods	13		MTR fuel
	4		Convair reactor fuel
U^{235}-Al fuel plates	330	2.9	North American Aviation, Inc.
	64		Argonne National Laboratory
Spent LITR fuel assemblies	24	2.96	Recovery of material at Idaho
Spent LITR control rods	4		Recovery of material at Idaho
Used BSR fuel assembly	1		Recovery of material at Idaho
Plutonium nitrate solution		1.29	Los Alamos
Depleted U-UNH solution		1,019	Harshaw Chemical Co.
Depleted U-UNH solution from metal recovery		85,423	Mallinckrodt Chemical Works and Y-12

TABLE 14. COMPARATIVE SUMMARY OF SF MATERIAL ACTIVITY FOR CY 1952 AND CY 1953

Quantities are expressed in kg of total SF content

TYPE OF MATERIAL	INVENTORY AS OF JAN. 1	MATERIAL RECEIVED OR PRODUCED	MATERIAL SHIPPED OR REMOVED	INVENTORY AS OF DEC. 31	MATERIAL UNACCOUNTED FOR
Enriched uranium, <75% U^{235}					
1952	1.42	2.61	0.81	3.22	0
1953	3.22	0.02	1.20	2.04	0
Enriched uranium, >75% U^{235}					
1952	32.01	61.80	61.56	31.98	0.27
1953	31.98	107.30	90.88	47.71	0.69
Normal uranium					
1952	85,960.57	95,448.08	103,238.08	78,132.47	37.83
1953	78,132.47	2,326.41	7,503.84	72,914.45	40.59
Depleted uranium					
1952	175,442.10	63,121.06	2,500.45	236,043.08	19.63
1953	236,043.08	15,847.57	86,465.45	165,420.25	4.95
Thorium					
1952	4,832.94	10,460.07	196.45	15,051.54	45.02
1953	15,051.54	5,709.05	83.22	20,664.95	12.42
Plutonium					
1952	6.74	9.40	4.51	11.52	0.11
1953	11.52	0.99	1.77	10.69	0.05
Uranium-233					
1952	0	3.10	2.45	0.65	0
1953	0.65	4.28	2.72	2.21	0

procedures contained provisions for using the economic value as the strategic value and also contained methods by which discards could be effected, it was necessary to obtain special permission to make discards. The adoption of a method termed "marginal cost data" as a guide in determining whether to recover or discard material allows the contractor more latitude in determining what materials should be discarded. This method was immediately adopted at ORNL.

All reported SF material losses and unaccounted-for quantities were investigated and recommendations were made to reduce them. All such losses were satisfactorily explained. Unaccounted-for losses of depleted uranium and thorium were reduced by a factor of 4, compared with 1952 losses. Plutonium losses were reduced by a factor of 2. Enriched- and normal-uranium unaccounted-for quantities showed increases over the previous year. The enriched-uranium loss increase was due to the second HRE leak, which occurred in November 1952, the amount of loss being unknown until late 1953. The loss (0.63 kg) was reported in 1953. The normal-uranium loss increase was due to a fire in an annealing furnace during 1952, the amount of loss being unknown until September 1953, at which time the loss (33 kg) was removed from accounting records.

Owing to the difficulties experienced at the HRE facility, several studies and surveys were made at the site. This entailed a review of records and of operating and measurement data. Following the successful operation of the reactor at the desired power level, measurement and critical data indicated a possible loss of approximately 1 kg of U^{235}. This indicated that a loss of 300 to 400 g of U^{235}, which was in addition to the 500 to 700 g involved in the second leak, had occurred; however, it was

determined that the volume of solution was essentially unchanged. It was decided to treat the system with a strong acid solution and reinventory the material. This yielded an additional 350 g of U^{235}. Analysis of oxide film recovered from the inside wall of a small segment of the reactor piping reflected a uranium content of 3 to 5 mg/cm^2, which could account for a rather large portion of the "unaccounted-for" loss.

Considerable time was spent in aiding Laboratory personnel to establish records on new programs and processes and in reviewing records for the purpose of simplifying them. The modified inventory and calculation sheet, Form X-333, seemed to cause some difficulty relative to filling in the required information. Personnel experiencing such difficulty were briefed on the form and given aid in completing inventory data.

The SF Accountability Group, in conjunction with the Research Shops, initiated a program of repairing, testing, and calibrating scales and balances used for measuring SF materials. This program had been recommended by the AEC SF Accountability Survey Group.