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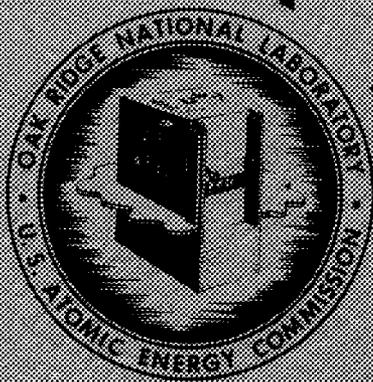
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RADIOISOTOPE PRODUCTION AND PROCESS DEVELOPMENT

ANNUAL REPORT FOR 1955

A. F. Rupp



OAK RIDGE NATIONAL LABORATORY

OPERATED BY

UNION CARBIDE NUCLEAR COMPANY

A Division of Union Carbide and Carbon Corporation

1956

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RADIOISOTOPE PRODUCTION AND PROCESS DEVELOPMENT

ANNUAL REPORT FOR 1955

By
A. F. Rupp

Compiled from Data by:
E. E. Beauchamp
J. H. Gillette
E. J. Witkowski

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RADIOISOTOPE PRODUCTION AND PROCESS DEVELOPMENT ANNUAL REPORT FOR 1955

A. F. Rupp

RADIOISOTOPE PROGRAM SUMMARY

This report summarizes the main radioisotope production and process development activities during the calendar year 1955. The number of shipments made (12,749) showed only a slight increase over CY-1954 (12,585), which also was not much greater than CY-1953 (12,036). The total amount of

radioactivity shipped, however, has risen rapidly – from 10,587 curies in CY-1953 to 28,879 curies in CY-1954 and to 49,383 curies during the past year. The increase has been the result of a trend to larger quantities per package, or "wholesale business," and of increases in shipments of Co⁶⁰ and Cs¹³⁷ for large irradiation units. Details are given in Tables 1 through 4. Service irradiations in the reactors increased slightly, from 746 in CY-1954 to 785 this year; unprocessed irradiation units increased from 1734 to 2010. Gross income increased 6%, from \$1,594,901 to \$1,690,871.

The processed cyclotron program was discontinued on December 1, 1955, to allow commercial laboratories to take over that field; however, 94 service irradiations were made in the 86-in. cyclotron, and this part of the program is to be continued.

Activation analyses increased from 31 in CY-1954 to 40 this year, with a total income of \$18,030; however, the income was still not sufficient to cover costs.

A new, 177-page, loose-leaf radioisotope catalog was compiled and printed; distribution of over 5000 copies was started at the end of the year.

Beginning July 1, 1955, cards were set up on the IBM system to keep records of all radioisotope transactions. Included in these compilations are data on amount and kind of radioisotopes shipped

TABLE 1. RADIOISOTOPE SALES DISTRIBUTION

	Calendar Year	
	1955	1954
Foreign	\$ 87,360	\$ 52,770
Project	63,320	183,072
Domestic	1,323,504	1,039,768
Cancer Program	196,246	251,190
Subsidy*		
Technical Co-operation Program	777	4,233
Local Sales to Carbide	19,349	63,498
Civilian Defense	315	370
Total	\$1,690,871	\$1,594,901**

*Income subsidized by the AEC. Does not include cash received.

**Includes project transfers amounting to \$7273.

TABLE 2. SALES AND INCOME OF MAJOR CATEGORIES

	Calendar Year 1955		
	Income	Shipments	Amount (mc)
Carbon-14	\$ 193,500	226	5,595
Cesium-137		121	2,031,908
Cobalt-60	260,077	431	22,513,201
Cobalt-60 (teletherapy)	163,730	19	19,569,000
Iodine-131	517,526	4,965	668,578
Phosphorus-32	192,738	2,504	158,124
Others	393,657	4,483	4,436,588
Total Radioisotopes	\$1,721,228	12,749	49,382,994
Boron	\$ 30,806		11,519 g
Helium-3			6,156 cc

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TABLE 3. RADIOISOTOPE SALES

HSA – High specific activity

LSA – Low specific activity

CF – Carrier-free

E – Enriched target

Product	Quantity Sold (mc)	
	1955	1954
Antimony-124, solution, HSA	278	262
Antimony-Be, neutron sources (9)	29,000	
Antimony-125, solution, CF	5	3
Argon-37, gas (5 ampoules)	11	
Arsenic-73, -74, solution, CF		2
Barium-133, solution, HSA	9	6
Barium-140, solution, CF	933	819
Beryllium-7, solution, CF	7	24
Bismuth-210, solution, HSA	16	
Cadmium-115, solution, HSA	21	15
Cadmium-115m, solution, HSA	8	
Calcium-45, solution, all grades		1,811
Calcium-45, solution, LSA	453	
Calcium-45, solution, HSA	1,643	
Calcium-45, solution, E	70	
Calcium-45, solution, CF	13 (μ c)	
Carbon-14, dry BaCO ₃ , HSA	5,595	4,767
Carbon-14, organic compounds	187	216
Cerium-141, solution, CF	235	39
Cerium-144, solution, CF	1,001	2,927
Cesium-134, solution, HSA	2,366	2,001
Cesium-137, solution, CF	34,608	327,368
Cesium-137, pelleted CsCl (92 sources)	1,997,000	
Chlorine-36, solution, HSA	4	2
Chromium-51, solution, HSA	7,570	4,947
Chromium-51, solution, E	65	
Cobalt-57, solution, CF	23	9
Cobalt-58, solution, CF	2	13
Cobalt-60, solution, HSA	1,401	6,012
Cobalt-60 metal (778 sources)	42,081,000	
Europium-155, solution	22	21
Fission products, solution	1,332	1,326
Gold-198, solution, HSA	305	
Hydrogen-3, gas (105 ampoules)	512,309	
Hydrogen-3-zirconium (89 targets)	80,000	
Indium-111, solution, CF		3
Indium-114, solution, HSA	65	52
Iodine-125, solution, CF		1
Iodine-129, solution	4 (mg)	
Iodine-131, solution, CF	668,578	557,482
Iridium-192, solution, HSA	368	12
Iridium-192 metal (297 sources)	3,559,000	

TABLE 3 (continued)

Product	Quantity Sold (mc)	
	1955	1954
Iron-55-59, solution, HSA	60	67
Iron-55, solution, E	104	115
Iron-55 metal (7 sources)	485	
Iron-59, solution, E	489	565
Krypton-85, gas, CF	65,501	2,831
Manganese-54, solution, CF	22	15
Mercury-203, solution, HSA	877	393
Neodymium-147, solution, CF	3	9
Nickel-63, solution, HSA	76	58
Niobium-95, solution, CF	47	68
Phosphorus-32, solution, HSA	157,456	152,922
Phosphorus-32, solution, CF	668	
Potassium-42, solution, HSA	302	40
Praseodymium-143, solution, CF	1	1
Promethium-147, solution, CF	5,697	218
Rubidium-86, solution, HSA	628	
Ruthenium-103, solution, CF	209	9
Ruthenium-106, solution, CF	717	1,012
Scandium-46, solution, HSA	167	62
Selenium-75, solution, HSA	420	359
Silver-110, solution, HSA	672	714
Sodium-22, solution	59	90
Sodium-24, solution, HSA	1,676	1,275
Strontium-85, solution, CF	12	5
Strontium-89, solution, CF	1,924	1,287
Strontium-90, solution, CF	129,455	116,536
Strontium-90, SrF ₂ glaze (12 sources)	9,000	
Sulfur-35, all types		11,766
Sulfur-35, solution (sulfate), CF	14,733	
Sulfur-35, solution (sulfide), HSA	322	
Sulfur-35, solution (element), HSA	980	
Tantalum-182, solution, HSA	75	13
Technetium-99, solution	1	2
Thallium-204, solution, HSA	1,712	2,258
Tin-113, solution, HSA	118	15
Tungsten-187, solution, HSA	126	162
Yttrium-88, solution, CF		2
Yttrium-91, solution, CF	250	390
Zinc-65, solution, HSA	1,640	762
Zinc-65, solution, CF	13	74
Zirconium-95, solution, CF	1,110	1,108
Helium-3, gas (23 ampoules)	5,195 (cc)	
Deuterium (6 targets)	6 (cc)	
Deuterium-zirconium (1 target)	1 (cc)	

TABLE 4. IRRADIATIONS IN OTHER REACTORS

	Calendar Year	
	1955	1954
At Hanford		
Antimony	2	4
Antimony metal	1	
Calcium carbonate		16
Ceramic materials		3
Cesium carbonate		1
Cesium chloride	2	2
Cobalt		10
Iridium		1
Iron-54		2
Iron oxide	7	8
Mercury	3	3
Nickel		3
Potassium chloride	32	16
Samarium		2
Selenium		1
Silver nitrate		1
Tantalum foil	2	
Thallium		2
Tin		2
Tungsten	3	2
Tungsten oxide	1	
Zinc		3
At the MTR		
Barium chloride	3	None
Cesium chloride	3	
Cobalt	18	
Nickel	5	
Silver metal	2	
Tin	5	
Zinc	5	

and on all production and distribution costs. This rather complete cost accounting system will provide the most detailed information yet compiled on radioisotope production costs.

Price reductions were made on several radioisotopes, most notably Cs¹³⁷, which was lowered from \$500 per curie to \$10 per curie, and Pm¹⁴⁷, lowered from \$500 per curie to \$100 per curie. The stepwise pricing of a number of short-lived fission products was eliminated, and the prices were slightly increased to more nearly cover production costs of these items, which have low sales volume.

Twenty-nine new, short-half-life products, ranging from Sb¹²² to Y⁹⁰, were offered for sale as the result of increased hot cell space and faster processing techniques. Most of these products are made in the LITR, which provides the customer with material that has a higher specific activity, has been assayed, and is in solution form.

The C¹⁴ organic compounds remaining in stock were sold to the highest bidders, and the account was closed out. Commercial laboratories now supply all C¹⁴ organic compounds.

Major development items were: improved techniques for handling, assaying, and fabricating sources of over 10,000 curies of Co⁶⁰ per batch; separation of Am²⁴¹ from Pm¹⁴⁷ by ion-exchange methods; design of new, more efficient isotope shipping containers; separation of Xe^{131m} from I¹³¹; transportation of 5000-curie batches of high-level radioactive waste from the ICPP; semi-works testing of flowsheets to be used in the Fission-Product Pilot Plant; and completion of design phases of the Fission-Product Pilot Plant.

IODINE-131

Iodine-131 continued to lead in the number of shipments made. The total sales for 1955 (668,578) represent a 20% increase over the previous year. The cost per millicurie sold was reduced from 30¢ to 25¢. This cost decrease was made possible by the excellent performance of the new equipment put into operation in 1954. Repairs requiring decontamination of the shielded equipment were necessary on only three occasions, but they were accomplished with relative ease and at comparatively low cost.

PHOSPHORUS-32

The demand in 1955 for P³², which is second in sales volume, increased 3.4% over that in 1954, to 158,124 mc, while the over-all production cost was reduced 5.6%. The total cost per millicurie of P³² sold was reduced from 92¢ to 84¢. The operation of this process was almost trouble-free. New extractors and hood, which were installed in 1954, performed satisfactorily, substantially reduced the radiation exposure to operators, and required practically no maintenance.

One hundred grams of sulfur was irradiated one week in an LITR half-fuel element to determine the yield of P³². The yield was approximately 100 mc per gram of sulfur, so that with the usual six-week irradiation period, roughly 300 times

more P^{32} per gram of sulfur can be made in the LITR than in the ORNL Graphite Reactor; even better yields will be obtained in the ORR. This will allow a great simplification of P^{32} processing. Irradiation cans and a holder for six of the cans were designed for irradiating sulfur in a half-fuel element. Since the necessary production can be achieved with a few hundred grams of sulfur, a small vacuum-distillation apparatus is being developed to produce P^{32} by distilling off the sulfur, leaving the P^{32} behind.

CARBON-14

Two hundred cans of beryllium nitride were prepared for irradiation at Hanford. Since the new cans will no longer have to be melted, the unprocessed material in inventory was dejacketed, and the furnace used for jacket removal was dismantled. A report was issued (ORNL-1962) which describes the production of C^{14} by the Be_3N_2 process.

The sale of carbon-labeled compounds held in inventory was completed. The sale was conducted on a bid basis, and the total proceeds amounted to approximately \$5000.

COBALT-60

Sales

The cobalt distribution program continued at an accelerated pace, with most of the cobalt being allotted to the teletherapy program. However, all the lower specific activity material (<10 curies/g) was allocated to orders for irradiation units; the largest order shipped totaled 10,000 curies. While more orders were received for high-specific-activity sources (>40 curies/g) for teletherapy than could be filled from current production, the backlog of orders was not increased during the year. The supply of cobalt from the MTR and HEW was still inadequate in relation to the total number of requests received for this material. Additional cobalt has been sent to both these sites and to Savannah River for irradiation.

New Methods of Handling Cobalt

In the past, irradiated cobalt from the Arco and Hanford reactors has been received at the canal at Building 3001, and the cans have been opened under water. This method of slug opening was both difficult and hazardous, and often resulted in exposure of personnel to radiation; an additional

problem was the resulting contamination of the canal water. The operation is now performed in the new 10,000-curie manipulator cell (MKC), and a modified power hack saw is used for opening the slugs. The opening, assaying, and storing of the irradiated material are now routine operations.

Cobalt Storage Facility and Irradiator

A combined underground storage facility and irradiator has been designed for storing approximately 300,000 curies of Co^{60} ; the facility is 6 ft square and $8\frac{1}{2}$ ft deep and consists of 92 stainless steel, $1\frac{1}{4}$ -in.-dia tubes arranged around a 1-ft square shaft. The central shaft hole contains a shielding plug that is 1 ft square and $7\frac{1}{2}$ ft long, with a 1-ft³ opening at the bottom where specimens may be introduced. The shielding plug is lifted by a portable hoist, and a safety device is used to lock the plug in the "up" position. The shield is a combination of barytes concrete and lead; the individual storage tube plugs are filled with lead. Provisions have been made for circulating cooling gases around the tubes; air flow will be automatically controlled to provide uniform temperature. The storage tubes and shaft are made as a complete unit, and the barytes concrete will be poured around it in three steps. Lead shot and bricks will be used for the top shield. The storage cans will be fabricated from aluminum with chrome-plated cold-rolled steel caps so that they can be picked up with a magnet. This facility will be completed in March 1956.

Multi-Kilocurie Cell

Construction of the 10,000-curie-capacity remote manipulator cell (MKC), shown in Fig. 1, was completed, and full-scale use of the cell was begun. The MKC has 3-ft-thick walls of barytes concrete and is lined with stainless steel. The cell is equipped with a Corning high-density-glass shielding window, Model 8 manipulators, a 1-ton bridge crane operated by compressed air motors, and two track-mounted shielding doors which allow the entire end of the cell to be opened. While the designed capacity of the cell is 10,000 curies of Co^{60} , the largest source loaded to date, which was in excess of this amount, produced no measurable amount of radiation in the operating area.

With the addition of this cell to existing facilities, scheduling problems have been greatly simplified, and the exposures of personnel to radiation have fallen to an all-time low. The decrease in



Fig. 1. Multi-Kilocurie Manipulator Cell (MKC) for Handling Amounts of Radioactivity in Excess of 10,000 curies of Co^{60} , or Equivalent.

exposures and the corresponding increase in allowable working time permit greater emphasis on the improvement of techniques and workmanship.

Cobalt Protective Coatings

Experiments indicate that cobalt metal diffuses through electroplated layers of gold, nickel, or copper at rates that are dependent upon the temperature. Of the plates tested, the nickel plate was the most satisfactory in minimizing the diffusion through the plated surface at the high temperatures experienced during MTR irradiations. Newer can designs are directed toward increasing the heat transfer from the cobalt pieces to obtain lower temperatures.

Cobalt Melting and Casting

It was demonstrated that induction heating can be used for melting and molding radioactive cobalt

metal pellets by remote control. A run was made in which a 1×1 cm "cold" cobalt pellet was melted and allowed to pour into a quartz mold in an air pressure of approximately 5μ . Approximately 1% of the cobalt metal evaporated and condensed immediately above the liquid during the melting process. Further work indicated that losses due to evaporation could be reduced substantially by melting the cobalt in a helium atmosphere at approximately 750 mm pressure.

CESIUM-137

Cesium production was continued from semi-works operations this year. Almost all the material produced went into inventory to be used for teletherapy sources or was used to fabricate radiographic-type sealed sources. The radiographic sources were made in sizes ranging from 5 to 100 curies. They were fabricated by pelleting pure

fission-product CsCl and sealing the pellets in a double-walled stainless steel capsule. It appears that these sources will be quite popular for radiography, as gamma-ray standards, and as sources of radiation for laboratory work. Eighty-nine small sources, totaling 839 curies of Cs¹³⁷, were made during one series of operations.

The 1540-curie Cs¹³⁷ teletherapy source made for ORINS two years ago was loaded into their machine, and a series of tests has been started to evaluate the machine. During the two-year storage period, no signs of deterioration of the source were noted.

A description of further chemical process development on cesium will be found under the Fission-Product Pilot Plant section.

STRONTIUM-90

A steady increase in the demand for Sr⁹⁰ was experienced, but production is still on a fairly small scale. No difficulty was experienced in supplying the demand.

Concentrated solutions from the Fission-Products Semi-Works containing Sr⁹⁰ were further processed to obtain relatively pure Sr⁹⁰. Both Sr⁹⁰ and inactive barium were precipitated from 83% nitric acid solution to remove iron, sodium, chromium, and potassium from the alkaline earths. Inactive fission-product barium was precipitated from cold 12 N HCl solution to separate it from strontium.

Approximately 135 curies of Sr⁹⁰ was purified during 1955. Strontium-90 left in the 83% nitric acid solution was not considered to be recoverable; this was approximately 5% of the total amount that was processed.

Equipment used for the purification was installed in the remote manipulator cell in Building 3030. Solutions handled were of concentrations ranging from 0.2 to 0.4 curie/ml. After the strontium was processed, the equipment was dismantled and the cell was readily decontaminated, thus demonstrating the feasibility of conducting such operations in manipulator cells.

PROMETHIUM-147

Promethium is effectively separated from europium by ion exchange, but the long-lived alpha emitter Am²⁴¹ remains with the promethium. A study of the distribution coefficients of the promethium and americium in H₂SiF₆ and in equilibrium with Nalcite HCR, a 100- to 200-mesh ion-exchange resin, indicated that these two nuclides could be separated

by ion exchange by eluting from the resin bed with 2.0 M hydrofluosilicic acid (see Fig. 2). This method was tested first with a small amount of promethium and later with approximately 30 curies of activity. The results of the large-scale run are given in Fig. 3. To recover the promethium from

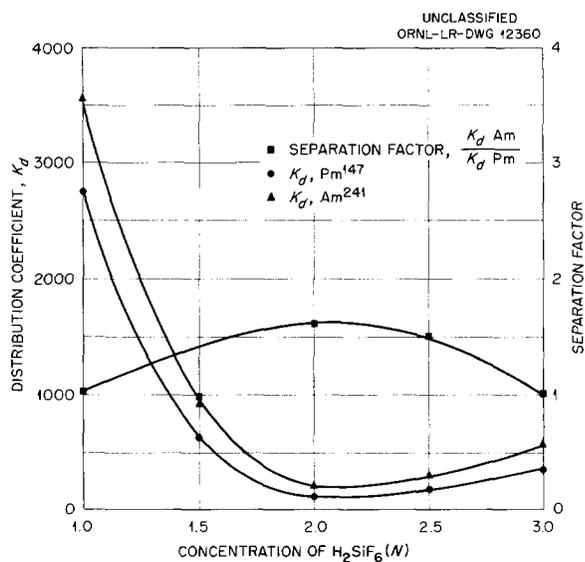


Fig. 2. Equilibrium Studies of Am²⁴¹ and Pm¹⁴⁷ in H₂SiF₆ Solution. Agitated with Nalcite HCR resin.

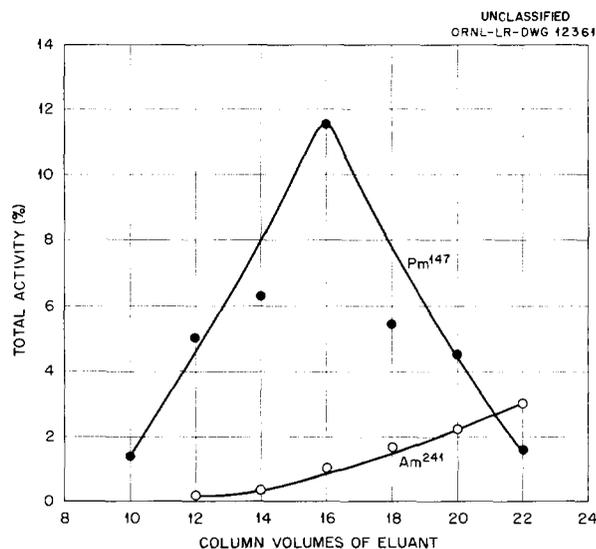


Fig. 3. Elution of Pm¹⁴⁷ and Am²⁴¹ from Nalcite HCR Resin. Conditions: temperature, 30°C; mesh size of resin, 100 to 200; elutrient, 2.0 M H₂SiF₆.

the eluant, it was adsorbed on a small cationic resin bed after the 2.0 M H_2SiF_6 eluant from the column was diluted to 1.0 M. The promethium was then removed from this relatively small amount of ion-exchange resin by elution with 6 N HCl, giving a Pm^{147} product relatively free of Am^{241} .

RADIOISOTOPE SHIPPING

New Containers

At the beginning of the radioisotope distribution program in 1946, all shipments were made in comparatively heavy returnable containers; a continuing effort has been made to develop new containers that would reduce the shipping costs, which are borne by the customers. By the end of 1954 this had been accomplished mainly by the substitution of cheap, disposable, light containers in the form of fiberboard boxes, tin cans, and other relatively inexpensive materials readily available on the market. At the present time, approximately 90% of the shipments are made in disposable containers.

The returnable containers used for the remaining 10% of the shipments are too heavily shielded to be economically replaced by disposable containers. Therefore a design study was made during 1955 on reducing their weight. The results indicated that a sizable reduction in weight (in some cases as high as 25%) could be accomplished on some of the containers used for shipment of solid materials by substituting reinforced wooden pallets and blocks for the outside wooden boxes. A design was submitted to the Bureau of Explosives and was approved, and the necessary container alterations are now being made.

A new outside container made of aluminum and wood was also designed to replace the larger and heavier wooden boxes now used for certain kinds of shipments. The new design not only reduces the weight of the packages but also provides an additional seal to make the containers safer. The design has been submitted to the Bureau of Explosives and is now awaiting formal approval.

Calculations have shown that approximately 50% of the weight of the very heavy containers (ranging from 250 to 6000 lb) could be eliminated by using depleted uranium instead of lead for the shielding. The AEC has agreed to an allocation of a quantity of depleted uranium for this purpose, with the restriction that the containers be used only for project shipments. Four of the smaller type of containers have been fabricated but have not yet been put into service.

Package Sealing

A machine that is expected to reduce the handling of the disposable lead containers during the sealing operation has been designed and partly assembled. The primary purpose of the machine is to reduce the exposure of operators to radiation. This machine will make it possible to substitute an O-ring gasket for the presently used tape, which will make the containers more nearly leakproof.

EUROPIUM-152, -154

The neutron activation of natural europium to produce Eu^{152} and Eu^{154} was carried over from the previous year. The buildup of activity was followed by measuring the radiation intensity of 345-mg Eu_2O_3 pellets after known intervals of neutron exposure. It now appears more certain that a saturation value of approximately 7 r/hr/meter (rhm) will be reached after a neutron exposure time of about 24 to 30 months at an average neutron flux of 1.3×10^{13} ; this would correspond to approximately 20 rhm for 1 g of Eu_2O_3 .

Comparative radiation intensity measurements were made on one of the Eu_2O_3 pellets which had received 567 days of neutron activation in the LITR and on a Co^{60} wafer (1.4 g of cobalt) which contained 4 curies of activity. The results were as follows:

	Absorber	Intensity (rhm)
$Eu^{152, 154}$	$\frac{1}{4}$ in. of Al	6.0
	$\frac{1}{2}$ in. of Pb	3.0
Co^{60}	$\frac{1}{4}$ in. of Al	5.5
	$\frac{1}{2}$ in. of Pb	3.2

Since the gamma-intensity measurements were approximately equal for the two activities, it is reasonable to compare the neutron exposure time necessary to produce the above activities. In the same neutron flux, the Co^{60} activity would be produced in $3\frac{1}{4}$ months, whereas about 19 months would be required for the europium activation.

The difference in half life – about 15 years for $Eu^{152, 154}$ and 5.3 years for Co^{60} – would not compensate for the sixfold greater activation time required for the europium activation. It seems unlikely that europium gamma-ray sources will compete with Co^{60} except for special applications.

RADIOACTIVE GASES

Krypton-85

A cell and the process equipment for the production of fission-product Kr^{85} were completed and placed in operation. The equipment is also designed for the recovery of fission-product xenon. Xenon and other gases which are condensable from the raw gases are removed by use of a cold trap at -195°C . The krypton is then concentrated by adsorption on a charcoal column at -80°C . The concentrated xenon and krypton are further purified by reacting the impurities with calcium metal and by eluting from a charcoal column in a stream of helium. Approximately 200 curies of Kr^{85} was purified in 1955.

Xenon-131m

A procedure was developed for the production of Xe^{131m} . The iodine from which the Xe^{131m} as a daughter product is obtained is precipitated with iodine carrier as silver iodide. After the silver iodide is filtered onto a microfilter, it is placed in a nickel tube with a small quantity of calcium metal; then the tube is evacuated. After six days (to allow growth of the 12-day Xe^{131m}) the tube and contents are heated to 1000°C . The Xe^{131m} is pumped off and then purified by passage over calcium metal.

Argon-37

Five curies of carrier-free A^{37} was produced for experimental work by the Physics Division. Irradiated CaO (from the LITR) was dissolved, and the gases released were collected in a nickel tube containing calcium metal. When heated, the calcium metal reacted with all contaminants, allowing pure A^{37} to be pumped off.

Tritium-Zirconium Targets

Several important developments occurred during the year which resulted in the preparation of better Zr-H^3 targets. A clearer understanding of the mechanism of the process reduced the number of rejections and thus lowered the unit cost of the targets.

Quality standards which are more stringent than those previously used were adopted. An acceptable target now must pass a visual inspection for good adhesion between the zirconium and backing and for proper metallic luster of the impregnated zirconium and must pass a radiometric inspection for tritium uptake. In order to prepare targets that

would consistently pass these tests, several phases of the procedure were reinvestigated.

A ceramic heater was devised for heating the targets during impregnation, which resulted in more uniform heating with better temperature control. Temperature studies were made for determining the optimum impregnation conditions. These studies, which are still in progress, have demonstrated that the formation of the "nitride film" which occurs during impregnation is dependent on time and temperature.

When copper was used as the backing material, an acceptable target could not be prepared. Since the use of copper is reported in the literature, this problem received intensive study. It was found that under impregnation conditions the zirconium diffused into the copper, resulting in an alloy which would not take up tritium. This phenomenon was found to exist with many of the metals which were used as backings; therefore, targets are now prepared only on those metals which experience has shown to be satisfactory.

As a corollary to the investigation of backing metals, the problem of degassing the backing was investigated. It was found that, contrary to some reports, all backing metals must be thoroughly degassed in order for successful Zr-H^3 targets to be prepared.

TRANSPORTATION OF RADIOACTIVE WASTE

Shielded Transfer Tank

Fabrication of a Shielded Transfer Tank (STT) (Fig. 4) was completed at a total cost of \$23,000, including the cost of revisions to improve its safety during the test period. The STT is a 250-gal-capacity, spherical, stainless steel tank, 4 ft in diameter, enclosed by a $5\frac{1}{2}$ -in.-thick lead shield and an outer shell of $\frac{13}{16}$ -in.-thick stainless-clad steel. The total weight is about 28,000 lb.

The STT was tested for shield integrity, pressure tested with 90 psi air, and tested under vacuum. After a test in which the tank was filled with fission-product solution from W-15 at ORNL and then emptied, the STT was shipped by rail to the Idaho Chemical Processing Plant (ICPP), where it was filled with Redox IAW waste solution. The tank was shipped back to ORNL and unloaded without incident. Radiation on the outside of the STT was 6.2 mr/hr, as measured with a Victoreen survey meter. The volume of the first shipment was 166 gal, containing 445 curies of Cs^{137} and about 5000 curies of total fission products. The second



Fig. 4. Shielded Transfer Tank (STT-1).

shipment was equally successful, containing approximately 210 gal of the same waste.

A pressure increase of 70 in. Hg had been estimated, based on factors usually employed in radiation chemistry, but it was found that the pressure increase in the closed container due to radiation decomposition of water was only a maximum of 3.3 in. Hg in 12 days.

ICPP Loading Station

Equipment for loading the STT at the ICPP was designed. The STT is filled by vacuum from a long cylindrical tank inserted in a riser in ICPP waste tank WM-180, into which fission-product solution is jettted continuously from the waste tank. A two-stage transfer was used because the depth of the liquid in the tank is too great for a single-stage transfer by vacuum.

Fabrication of the intermediate transfer tank was done by ORNL, and construction of the control

room and installation of the intermediate transfer tank were done by Phillips Petroleum Company. The equipment performed well in the loading of the STT.

FISSION-PRODUCT PILOT PLANT

Engineering and Construction

The design phase of the Fission-Product Pilot Plant (F3P) was completed during 1955 with the transmittal to the AEC of construction drawings and specifications for the building and cell block and with the fabrication and installation of process equipment.

The main part of the plant (Fig. 5) is housed in a concrete-block building and consists of a cell block of 17 cells and associated services, makeup areas, offices, instrument panel boards, and a 20-ton bridge crane. Four underground cells adjacent to the building will contain process storage and waste tanks.

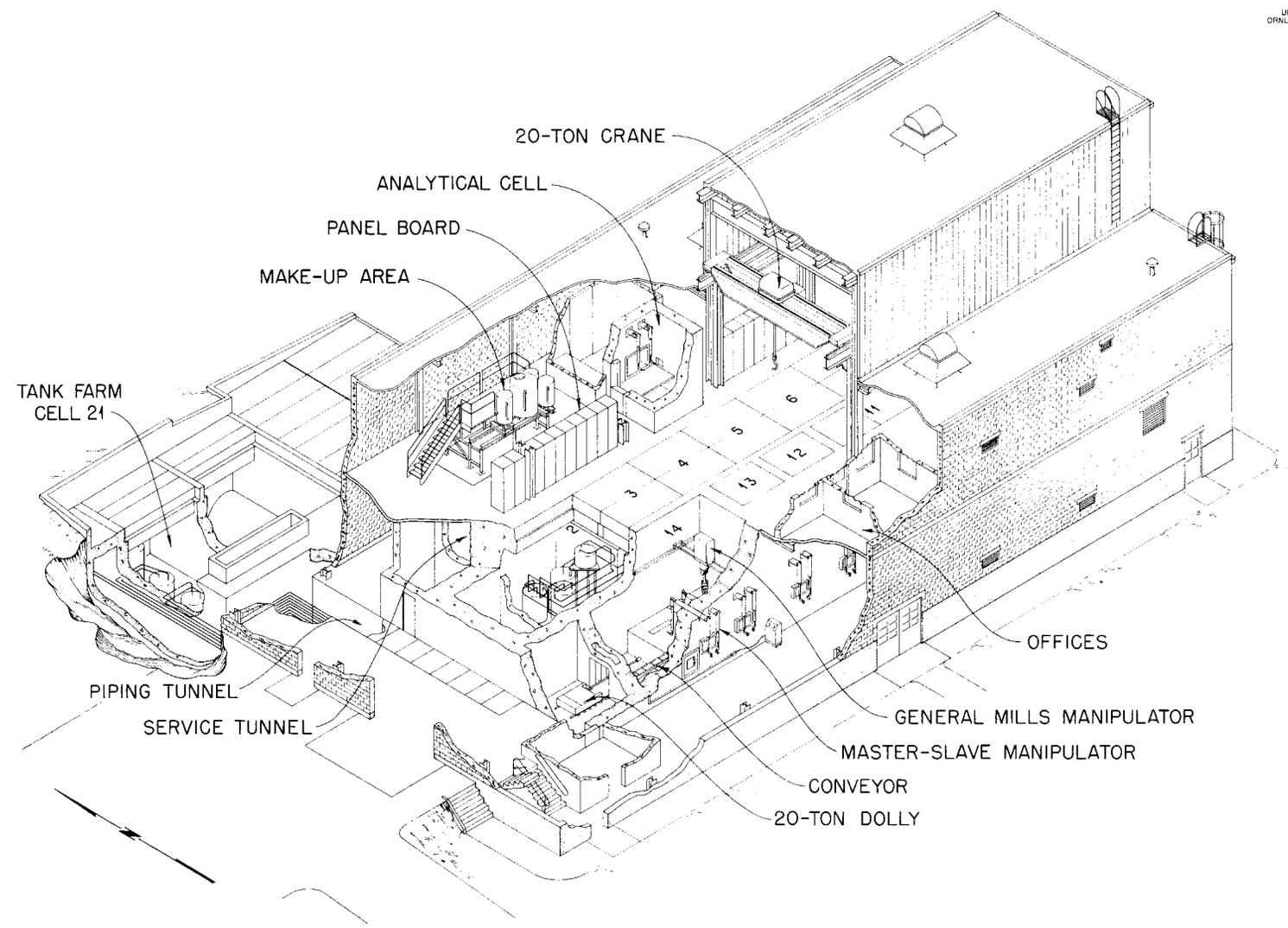


Fig. 5. Fission-Product Pilot Plant (F3P).

Process vessels and piping for chemical processing will be installed in ten cells, and manipulators and viewing windows will be installed in four cells for final processing of separated fission products and source fabrication. One cell will contain equipment for the decontamination of shielded carriers and process equipment. Space for future expansion is provided in two spare cells. Ventilation to the cell and hot off-gas to the vessels will be provided by underground ducts extending to the existing stack area.

Provisions were made so that condensate containing weak nitric acid from the feed evaporator can be pumped back to the Metal Recovery Plant for concentration and re-use.

A cell is located above the process cell bank and contains a pair of remote manipulators and will be used to take process samples. Two types of samplers will be used: a recirculating sampler, using the air-lift principle, will furnish 4- and 20-ml samples; a 1-liter sample vessel will be used to measure crystal volumes. The air-lift principle will be used to lift the 1-liter sample to the vessel, where it will be measured for crystal volume and from which it will flow back by gravity. Small samples may be taken from a three-way stopcock at the bottom of the 1-liter vessel. Provisions have been made for eleven recirculating samplers and four 1-liter samplers. Dilutions, pH measurements, titrations, and gross gamma readings may also be made in the cell.

Final processing of the source material and sealing it in containers will be done in manipulator cells. Seven pairs of Model 8 master slave manipulators will be used, including a pair in the analytical cell. A General Mills Model E mechanical arm will be used for heavy-duty manipulation in a cell where the source assemblies are handled and placed in the shipping containers. An inter-cell conveyor, 10 in. wide, will make it possible to transfer sources and equipment from one cell to another. Shielded transfer casks will be moved into the loading cell on a 20-ton motorized dolly. The casks will be lowered into the cell with the 20-ton overhead bridge crane.

A 2000-gal steel tank, with provisions for heating, will be located outside the building for the storage of commercial 50% NaOH solution. A 350-gal stainless steel tank will be used for storing nitric acid. Pumps will be used to transfer these solutions to the makeup areas. Nine stainless steel tanks, ranging in size from 50 to 250 gal,

will be installed in the makeup areas above the process cells. Addition of the solution to the process tanks will be by gravity flow, with a vent located in the line inside the cell to reduce the possibility of radioactivity being forced up into the makeup areas.

In order to expedite construction of the plant, the project was divided into three phases. Phase I, which includes the building, cell block, and tank farm, was designed by ORNL and the McPherson Company and was released for bids in June. Phase II, which includes process equipment, piping, and instrumentation fabrication and installation, was designed by ORNL and was released for bids in October. Phase III covers purchase and installation of specialized equipment, such as manipulators and viewing windows, by ORNL.

Award of the Phase I contract was made to the Malan Construction Company, who began construction on October 21, 1955. Prior to this date, the excavation for the building and cell block footings and for the ventilation ducts was done by ORNL because the area was found to be generally contaminated. After work was begun by the contractor, the tank farm cell area was flooded with contaminated water during heavy rains, which delayed progress of the project and necessitated much work by ORNL. Parts of the project completed by the contractor during the year include the following: pouring of concrete for the floor and for all but one wall of the tank farm cells, pouring of building footings, and laying of the compacted gravel backfill under the building area.

After bids were opened for Phase II construction in November, the low bid was found to be unreasonably in excess of the engineering estimate for this part of the project. Therefore, all bids were rejected, and it was decided that all process equipment would be purchased by ORNL and be installed by a cost-plus-fixed-fee contractor.

Purex Flowsheet Tests and Equipment Testing

The Purex waste processing flowsheet proposed for the F3P was tested on a semi-works scale (20-liter volume or $\frac{1}{40}$ plant scale) to determine the efficiency of precipitations, the volume of precipitates, the centrifugation characteristics of slurries, and the dissolution or slurring characteristics of the precipitate cake, and to develop correct techniques.

Each precipitation in the flowsheet was made separately; equivalent amounts of inactive chemi-

calcs were used in place of fission products, and the process was followed by means of an appropriate radioactive tracer. A single tracer was used in each experiment to simplify analysis. In general, it was found that on this scale the precipitations were at least 95% complete and that the precipitates formed could be separated by centrifugation in a solid bowl centrifuge at 825 times gravity and 4 min of residence time with a loss of about 3%.

In the course of the semi-works experiments, a technique of making controlled pH precipitations by sparging with anhydrous ammonia was developed. A corollary of these experiments was the development of a dependable mechanical system for withdrawing a representative sample of the reacting solution for pH measurement.

It was attempted to determine precipitation end points by the use of noble metal electrodes immersed in the solution, but this approach was proved to be impractical. A crystal-bed level finder using a vertically ranging dip tube was developed but was not included in the final plant design because of its mechanical complexity.

Wear tests were run on several automatic valves intended for plant use, and corrosion tests were made on various alloys in simulated plant solutions. Based on the results of the equipment tests and semi-works experiments, many new features were incorporated in the final plant design.

An experimental filter assembly was fabricated to obtain design data for the crystallizers. The filter was made of 200-mesh, stainless steel wire cloth in the form of a cylinder, 1½ in. in diameter and 24 in. long. A drawoff tube, ¾-in. NPS stainless steel pipe inside the filter cylinder, extended within ¼ in. of the bottom. A 3½-in.-dia tube was mounted on the outside of the filter so that air forced through the drawoff line would replace the solution in contact with the filter during the formation of the crystals. The 200-mesh wire cloth was supported with a backing of 14-mesh, stainless steel cloth. A ½-in. stainless steel jet supplied the suction to transfer the solution through the filter, and provisions were made so that steam and air could be forced back through the filter to clear the screen of crystals. The transfer rate from a solution containing 30% crystals was 6.5 liters/min.

Crystallizations were made with the solution in contact with the filter to determine the length of time necessary for the steam to remove the crystals

from the filter. The flow through the filter was 3 liters/min. Steam flowing through the filter cleared it of crystals in less than 3 min. The temperature rise of the solution was only 2°C.

Corrosion tests made on the 200-mesh stainless steel cloth under operating conditions, except for the presence of radiation, showed no loss of weight after 166 hr of exposure.

Fission-Product Semi-Works Development

Seven thousand curies of Cs¹³⁷ was recovered from fission-product wastes by use of the ammonium alum crystallization process. Three hundred curies of Sr⁹⁰, 500 curies of mixed rare earths, and small amounts of technetium were also produced.

The results of continued semi-works operation indicate the feasibility of utilizing precipitation reactions to recover fission products from wastes. Rare earths have been precipitated as hydroxides, oxalates, and phosphates, without the addition of carriers, in 100- and 1000-curie batches with high recoveries. Strontium carbonate and nitrate precipitations have been satisfactory from a yield standpoint. High radiation levels do not interfere with the crystallization of the cesium alum or subsequent precipitation separations.

The low yields of technetium experienced were probably due to the heat sensitivity or radiation sensitivity of the organic reagent used, tetraphenyl arsonium nitrate. The poor material balances obtained indicate the loss of organic precipitate. Laboratory results show that excellent recoveries of technetium are possible; however, these results have not been achieved in semi-works equipment.

Investigations were made on the sorption of technetium by strongly basic anion exchangers and on its precipitation as sulfide from acid solution or as tetraphenyl pertechnetate with tetraphenyl nitrate. It was noted that technetium does not precipitate as the hydroxide or carbonate as had previously been reported. This error in observation was probably caused by the mixing of iron hydroxide and phenyl arsonium pertechnetate in the semi-works equipment.

Cesium Process Control

A method based on the exothermic property of alum crystal formation was developed for following the course of Cs¹³⁷ purification by alum crystallizations. The equipment used was a sensitive thermocouple and a Brown potentiometer. Cooling

curves were recorded for solutions containing ammonium alum, cesium alum, and rubidium alum, either separately or in mixtures, covering the expected range of concentrations. A very definite break in the cooling curves for ammonium alum was observed over the concentration range of 400 to 100 g/liter. A smaller inflection in the cooling curves was also detectable at the cesium alum crystallization temperature over the concentration range of 10 to 50 g of cesium alum per liter of solution, which also contained ammonium alum. The concentrations of rubidium alum were too low to cause a detectable inflection. The concentration of ammonium alum may be determined within ± 10 g/liter by this method.

The order of decreasing solubility of the three alums present in the process is: ammonium, rubidium, and cesium. Initially, the ammonium alum concentration is from five to ten times greater than the cesium alum concentration and serves as a carrier for the cesium by the co-crystallization mechanism. Subsequent crystallizations reduce the ratio of ammonium to cesium alum, and the ammonium alum assumes the role of a salting agent for the cesium alum. As little as 50 g of ammonium alum per liter was sufficient to reduce the cesium alum solubility to less than 0.25 g/liter, while the rubidium alum solubility at this point was at least 3 g/liter. Therefore, it was possible to obtain essentially pure fission-product cesium by properly adjusting the ammonium alum concentration. The behavior of cesium and rubidium in the three-component system was determined with the aid of Cs^{137} and Rb^{86} radioisotopes. Once their behavior was established, the progress of the purification was followed by determining the ammonium alum concentration as described in the preceding paragraph.

A more comprehensive discussion of this technique, along with supporting data, was given in Appendix I of the interdivisional report for February 1955.

Conversion of Cs_2SO_4 to CsCl

The current process for converting Cs_2SO_4 to CsCl for source fabrication involves the use of an anion-exchange resin column to remove the sulfate ion. A review of all known methods for sulfate ion removal resulted in a decision to continue using the present method. An undesirable characteristic of ion-exchange resin-column operation is channeling of the solution through the

resin bed, which effectively reduces the total available capacity for ion exchange. A series of tests with columns up to 6 in. in diameter indicated that this problem may be largely avoided by operating the column in a backflow manner. The maximum backflow rate which should be used was ascertained to be 0.6 cm/min of linear flow. No movement of the resin was detected at this flow rate, and the maximum "tilt" of the band front on a 6-in.-dia column was about 1 in.

A column approximately 10 in. in diameter and 7 ft tall was designed for the F3P and will be operated by the backflow method.

Cesium-137 Pyrosulfate

Cesium pyrosulfate was tested as a possible cesium gamma-ray source compound. The salt melts at approximately 550°C . Since the melt is very corrosive when in contact with stainless steel and Monel metal, it may not be feasible to pour the molten salt into source containers; therefore this method would have no particular advantage over pressing cesium chloride into pellets.

Cesium Ferrocyanide

Selective precipitation of cesium on various ferrocyanide precipitates was confirmed. Wet methods of decomposing ferrocyanide slurries were investigated, since there is a possibility that the other sites may produce crude cesium ferrocyanide slurries which could be used as feed material for the F3P. However, the general conclusion was that the ferrocyanide method was not the best choice for processing cesium from wastes.

Ruthenium Chemistry

Several methods were investigated for the recovery of fission-product ruthenium which would not involve the distillation of RuO_4 . A preliminary concentration of ruthenium is made by co-precipitation with $\text{Fe}(\text{OH})_3$. The problem then becomes one of separating the ruthenium from a large excess of iron. The methods tried may be classified under the general terms of (1) electrodeposition, (2) solvent extraction, (3) co-precipitation, (4) precipitation, and (5) reduction to the metal (chemical).

The experiments were qualitative in nature and did not constitute an exhaustive study. It was noted that trivalent ruthenium was not extracted readily from a nitric acid solution by tributyl phosphate but that trivalent iron could be readily

extracted. The separation was not quantitative and represents a departure from the basic idea of using precipitation reactions in the Fission-Product Pilot Plant.

Of the precipitation methods investigated the only satisfactory one was that in which ruthenium was reduced to the metal by electrodeposition or by action of a metal such as magnesium or zinc. Although a recovery problem is created by the difficulty of dissolving ruthenium metal in acids compatible with stainless steel, the method may be useful in the decontamination of waste streams from the plant and merits further investigation.

Redox Wastes

Development work was carried out on chemical precipitation methods of recovering fission products from aluminum nitrate wastes. It was found that cesium and aluminum can be removed from solution by formation of the ammonium alum. Strontium and rare-earth fractions were precipitated at a high pH by the addition of hydroxide and carbonate, with the aluminum remaining in solution as the aluminate. The processing of this type of waste is complicated by the presence of mercury, and further work is being done on removal methods for this element.

Separation of Cerium-144

Fission-product rare-earth concentrates after three to four years of decay contain, approximately, 75% Ce¹⁴⁴, 24% Pm¹⁴⁷, and 4% Eu¹⁵⁵. The Ce¹⁴⁴ can be effectively separated from the other rare-earth activities and alpha contamination by oxidizing Ce³⁺ to Ce⁴⁺ and precipitating as Ce(IO₃)₄. Because of the presence of iron in the solution and so that the activity will be concentrated, the rare earths have to be precipitated as oxalates before the final precipitation of cerium as ceric iodate. Losses of Ce¹⁴⁴ into the supernatant liquid from the oxalate precipitation are approximately 1%, but on the iodate precipitation they are less than 1%.

The separation of cerium in the 4+ valence state from other rare earths was demonstrated by three different methods: precipitation of ceric iodate, precipitation of ceric pyrophosphate, and extraction of ceric nitrate into tributylphosphate from 6 N HNO₃.

OTHER ITEMS OF INTEREST

Boltaron Cell Lining

A cement-block shielded cell in Building 3030 was altered for use as a manipulator cell for intermediate-level (100 curies of Co⁶⁰, or equivalent) radiochemical processing. The cell has 2-ft-thick, normal concrete-block walls and is equipped with a zinc bromide window and Model 4 manipulators. An innovation was the use of unplasticized polyvinyl chloride (Boltaron) sheet as the cell liner. This plastic can be hot-air welded, and the welds can be easily tested with a high-voltage spark. Experience with the cell indicates that the plastic liner has better decontamination characteristics than stainless steel.

Heliarc Welding Device for Remote Operation

A Heliarc welding device for remote operation was designed. Containers up to 3 in. in diameter and 12 in. long may be welded at the end or side in a vertical or horizontal position. The machine will be fabricated in two portable units. One of the units will include the welding electrode and a variable-speed motor and positioning mechanism, which will be placed in a manipulator cell. The other part of the device will include the welding machine, the recorder, and the inert gas cylinder and controls mounted on a wheeled dolly. The device will be used for sealing stainless steel source containers filled with multicurie amounts of radioactive material.

Sodium-24 Radiographic Sources

Experimental NaCl pellets were made in order to determine whether a sufficiently compact Na²⁴Cl pellet could be made for radiographic work. A pellet 1 cm in diameter by 1.3 cm in height weighed 2.173 g (density, 2.05 g/cc), which would contain approximately 3 curies of Na²⁴ if irradiated in the LITR. This is slightly low for radiographic work, but an acceptable source can probably be made in the ORR.

Cobalt-57 Source

The fabrication of a Co⁵⁷ source for experimental use by the Medical Division of the Oak Ridge Institute of Nuclear Studies was completed. The Co⁵⁷ activity was produced in the ORNL 86-in. cyclotron by the nuclear reaction:



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Cobalt-57 decays with a half life of 270 days by positron emission to 1.1×10^{-7} sec Fe^{57m} , which decays to stable Fe^{57} with the emission of a 0.014-Mev x ray. The Co^{57} radionuclide was isolated from the target material and other trace amounts of impurities on a Dowex-1 anion exchange column. A total of 557 mc of Co^{57} was separated.

The source was prepared by electroplating the cobalt activity on a 0.235-in.-dia platinum disk from an ammonium hydroxide solution. The plate was examined under a microscope and appeared to be a hard, evenly distributed, barely visible layer formed on the platinum surface. Smears made on the active surface of the source were low in activity count, indicating a deposit of strong adhesion. Additional protection against corrosion was given the source by the evaporation of a 0.000006-in.-thick SiO_2 film over the active surface. Radiation intensity measurements were made and were found to be approximately 2.8 r/hr at 10 cm.

Cadmium-109 Source

A 100-mc Cd^{109} source was fabricated by plating the radiocadmium onto a 1-cm-dia platinum disk. The active surface of the source was covered with approximately 0.000004 in. of SiO_2 to provide more protection against corrosion. The x-ray output was measured and was found to be 2.1 r/hr at 3 cm. The source is being used for research by the ORINS Medical Division.

Silver-111

A 100-mg palladium sample was irradiated in the fast flux (2×10^{13} neutrons/sec/cm²) of the LITR for a period of 14 days to produce 2 mc of 7.6-day Ag^{111} . Approximately 3% of the silver activity was Ag^{110} , which was produced by n -gamma reaction on the silver impurity present in the palladium target. The separation of the silver from the palladium was achieved by use of the anion-exchange-resin technique.

Strontium-90 Alloys

A long-range investigation was begun for finding a method for Sr^{90} beta-source fabrication which would have a low attenuation of the beta rays and at the same time would confine the activity for safety in application.

The formation of an alloy between strontium and a light metal such as magnesium or aluminum may meet the desired requirements. A few preliminary experiments were made in which a strontium-aluminum alloy preparation was unsuccessful. The literature offers little help on this problem, and it is anticipated that considerable experimentation will be required to devise a practical method suited to remote control.

Contributions to Meetings

At the Fourth Symposium on Hot Laboratories and Equipment, sponsored by the American Nuclear Society, the following papers were given: "Radioactive Gas Processing Equipment," R. E. McHenry; "Special Features of New Radioisotope Processing Cells," E. Lamb; "Specialized Devices for Manipulator Cell Operation," B. F. Early.

At the AEC Classified Sanitary Engineering Conference, held at Cincinnati, Ohio, two papers were given: "Decontamination of Fission Product Wastes with Separation of Kilocurie Quantities of Cesium, Strontium, Rare Earths, and Ruthenium," S. J. Rimshaw; "Operating Experience in the Disposal of Radioactive Wastes in Open Pits," E. J. Witkowski.

At the ORINS Conference on Rare Earths in Biochemical and Medical Research, "Reactor-Produced Isotopes" was given by R. S. Pressly.

Two papers were presented by A. F. Rupp at the International Conference on Peaceful Uses of Atomic Energy, Geneva, Switzerland: "Large-Scale Production of Radioisotopes" and "Methods of Handling Multikilocurie Quantities of Radioactive Materials."