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FOLDER 66 RADIOISOTOPES

The Early Days of the Radioisotope Production Program

By A. F. Rupp and E. E. Beauchamp

A. F. Rupp, Director of the Oak Ridge National Laboratory (ORNL) Isotopes Development Center, has always been one of the strongest advocates of producing and marketing radioisotopes for peaceful uses. Mr. Rupp was graduated with a B.S. in chemical engineering from Purdue University and, after working in the chemical engineering field for several years, he joined the staff of the Manhattan Project University of Chicago Metallurgical Laboratory in 1943. Shortly thereafter he transferred to ORNL (at that time the Clinton Laboratories) and since then has been closely associated with design and engineering of many systems and processes for handling radioisotopes. His concepts for isotope processing, for waste management, and for control of contamination are in use throughout the world.

Edward E. Beauchamp has worked at Oak Ridge since 1945 when he performed fission-product analyses at Clinton Laboratories for E. I. du Pont de Nemours & Co., Inc. In 1946 he became Supervisor of the Radioisotopes Process Development Department, and in 1952 he became Superintendent of the Radioisotopes Production and Process Development Department. After several years as Superintendent of the Isotopes Sales Department at Oak Ridge National Laboratory, Mr. Beauchamp was assigned to the Isotopes Division staff in 1966 as a research engineer involved in long-range programming.

By the end of World War II, a few radioisotopes were being produced routinely at the Manhattan Project Clinton Laboratories [later Oak Ridge National Laboratory (ORNL)] as an outgrowth of the work of experimenters who needed radioisotopes for their own research. Radioisotopes made in the Graphite Reactor, some of them processed (e.g., ^{14}C, ^{131}I), were also being distributed informally to workers at other Manhattan Project sites when the decision was reached in June 1946 to make radioisotopes routinely available to off-Project research workers throughout the country. A June 1946 article in *Science* (Fig. 1) announced the availability of radioisotopes to the scientific public. An order for 1 mc of ^{14}C placed by Barnard Free Skin and Cancer Hospital, St. Louis, Mo., was officially selected as the first to be filled on Aug. 2, 1946, thus launching the highly successful radioisotope program (Fig. 2). Some of the highlights of the early production are shown in Fig. 3.

The earliest support for the fledgling radioisotope program (for needs other than nuclear research) came from scientific research and the field of medicine. The stage had been set for this work by radioisotope pioneers who used cyclotron-produced materials for tracing biochemical pathways as an aid to medical diagnosis—radioiodine for thyroid studies, radiophosphorus for blood work, and radiocarbon for metabolic investigations. The relatively enormous capacity of the Graphite Reactor for the first time enabled production of quantities of radioisotopes large enough to allow hundreds of pent-up research projects to burst forth, filling the journals within a few years with much scientific and medical information. Now, after two decades, we note that radioisotopes have been used in almost every technical field, with current emphasis on radiography, industrial process radiation, and isotopic power.

At first, work on radioisotope production at Clinton Laboratories was scattered among chemists, physicists, and engineers in many sections of the Laboratory. The first ^{14}C, for example, was made by members of the physics and chemistry groups, using an ammonium nitrate solution circulating in a loop in the pile. (In retrospect, it is surprising to note that such an advanced technique as an in-pile circulating loop, though only moderately successful at the time, should have been used so early in the radioisotope program.) Other Laboratory groups developed methods for making ^{32}P from sulfur and ^{131}I from tellurium. However, the few chemically isolated radio-

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SCIENCE

Vol. 103, No. 2685 Friday, June 14, 1946

Availability of Radioactive Isotopes

Announcement From Headquarters, Manhattan Project, Washington, D.C.

PRODUCTION OF TRACER AND THERAPEUTIC RADIOISOTOPES has been heralded as one of the great peacetime contributions of the uranium chain-reacting pile. This use of the pile will unquestionably be rich in scientific, medical, and technological applications.

Manhattan Project scientific, technical, and administrative personnel have, since the inception of the pile, been cognizant of its peacetime potentialities and have, since the end of the war, been active in attempting to realize these opportunities. Since, however, war-built piles and wartime researches had other objectives, a considerable transition in researches, developments, and operations connected with piles must be effected before the supply of radioisotopes can begin to meet the demand.

AVAILABILITY OF RADIOISOTOPES

...e the extensive variety ...th the cyclotron beca... ...uch greater divers... ...ing projectil...

absorbed in the introduced material that the chain reaction ceases even though the control rods are withdrawn as far as feasible. With available pile facilities, this limit does not permit the production of a sufficient quantity and quality of many radioisotopes to meet anticipated national demands. To accomplish this it would very likely be necessary to build piles especially designed for the purpose.

(5) Technical problems involved in the irradiation of some materials have been, and will continue to be, responsible for delays in making certain isotopes available by routine irradiation. Examples of such problems are: (a) proper canning of the material to prevent rupture of the container by its internal action or by the external action of the coolant, with con-

Fig. 1 Reprint of first page of article from Science, *103: (1946) announcing availability of radioactive isotopes.*

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MONSANTO CHEMICAL COMPANY
CLINTON LABORATORIES
Oak Ridge, Tennessee

From J. A. Cox SHIPPING REQUEST Radioisotope Order #CL-7-3

To Isotope Shipping Dept.

Date August 1, 1946

Ship To
Dr. W. L. Simpson
Barnard Free Skin & Cancer Hospital (Research Dept
3427 Washington, St. Louis 3, Mo.
Bill To
Barnard Free Skin & Cancer Hospital (Research Dept
3427 Washington
St Louis, 3, Mo.
Spelling Billing Instructions

Customers Order # & Date
PO#842 July 22, 1946

Department No.

Route See Note

B/L No.

Container No. None

B/L Instructions

DESCRIPTION OF SHIPMENT

Quantity Ordered	Quantity Shipped	Material	Price	Extensions
1 mc C^{14}	1 mc C^{14}	____Box, weight____lbs.		
		Plus handling Plus Container Deposit Non-returnable container Container deposit will be refunded upon return of container, in good condition, to MCC - CL, Knoxville, Tenn. via, prepaid railway express.	None	None

Date Shipped: 8-2-46 Signed J. A. Cox
Isotopes Office

Shipped by: W. F. Stringfield
Isotope Shipping Department

Distribution: 1. Original to Accounting Dept.
2. Copy to Isotopes Office
3. Retained by Isotope Shipping Dept.
4. Retained by Traffic Dept
5. Producer

NOTE: Delivered to E. V. Cowdry representing Barnard Free Skin & Cancer Hospital 1 millicurie of C^{14}, August 2, 1946.

Received E. V. Cowdry
Barnard Free Skin & Can. Hosp.

Fig. 2 Document for first official shipment of radioisotopes after the Manhattan Project announcement in Science.

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isotopes were not as important at the time as the providing of irradiated "units" and reactor irradiation services. Selected target materials (e.g., sodium carbonate to produce ^{24}Na) were irradiated, as well as steel piston rings, seeds, petroleum, plastics, and a myriad of other objects from false teeth to diamonds. A simple, high-purity aluminum can (with a crimped lid) containing the target material was placed in a graphite "stringer," inserted into the pile, irradiated for days or months, and sent in a shielded container to the radioisotope user. Tools to open and handle the irradiated materials also were sent to the customer.

Shortly after the first ^{14}C shipment, all radioisotope production activities were consolidated and transferred to the reactor operating group; a development "group" (consisting of the two authors of this article) was also transferred in from one of the scientific divisions. Production, shipping, and development activities were quickly organized to provide, roughly in order of importance, the following:

1. Irradiation units and services
2. Carbon-14
3. Iodine-131
4. Phosphorus-32
5. Other separated radioisotopes

Scaled-Up Production

The group was faced with the problems of devising larger scale, improved production processes for the "Big 3" (^{14}C, ^{131}I, ^{32}P) and new processes for separating and purifying some 100 other isotopes. Some order of priority was needed, and first attention was given to selecting and preparing nitrogen-containing target material for ^{14}C production [first $Ca(NO_3)_2$ and later Be_3N_2] and to developing chemical processes for separating the ^{14}C. The chemical separation process basically called for target dissolution, volatilization by transformation to $^{14}CO_2$, absorption of the $^{14}CO_2$ in alkali, and finally precipitation as $Ba^{14}CO_3$. As so often happened, one of the most difficult problems turned out to be the apparently simple operation of removing the aluminum jackets from the irradiated slugs. Another difficulty was the rigid exclusion of atmospheric CO_2 at every stage of the process. After many trials with early-day remote-control tools such as saws and modified lathes, we finally melted off the jackets in an inert-atmosphere electric-tube furnace. Typical of these early days, this work was done in a field in the open air, using distance and time limitations for radiation control. Radiation instruments were relatively primitive, but, with this and similar operations, we were able to keep radiation exposure well within the permissible limits that were in effect at that time.

Physical facilities for the radioisotope program were practically nonexistent. Any hot-laboratory space, or indeed any laboratory space, tools, and instruments that could be "scrounged" were used. Often we felt that we were following too closely in Madame Curie's footsteps! Nevertheless, this early period was challenging, exciting, and highly productive; new processes were quickly devised for ^{35}S, ^{45}Ca, ^{59}Fe, most of the important fission products, etc. By 1948 more than 50 processes had been developed, and the basic design for an isotope production area had been drawn up. Construction of the ORNL Isotope Area was completed by the end of 1949, and we were sending out 6000 shipments per year of almost 100 different kinds of radioisotope preparations.

Early Equipment

Remote-control equipment was designed and fabricated at the Laboratory and put to use for the first time on a large scale in the Isotope Area; much of it (e.g., the radioisotope packing area) served with slight change for almost 20 years and is still in use today. "Over-the-barricade-with-mirrors" was the prevailing concept, but as cells with windows and manipulators were developed during the ensuing years, they were installed in the Isotope Area's individual, highly versatile process buildings. There was a great deal of preoccupation with remote-control devices during this early period because very little equipment of this kind had been built. Each hot-laboratory man had to create his own tools. The great advances in master–slave manipulators and associated hot-cell equipment, particularly by the remote-control group at Argonne National Laboratory (ANL), were still in the future. Later, when hot-laboratory devices and instruments became available commercially, radioisotope workers were able to turn their attention to their main job with highly increased efficiency. The modern hot cell changed radioisotope processing work; next to the high-flux reactors, it is the most important improvement that has come along in radioisotope production work.

Flux Intensity

It was evident from the first that the intensity of available neutron flux was of transcendent importance in radioisotope production. While the Graphite Reactor with its 5×10^{11} n/(cm^2)(sec) flux represented a mighty leap in the production capacity for radioactive materials, we had hardly started before we were wishing for 10 or 100 times that flux. Specific activities of short-lived radioisotopes were too low; hundreds of $Ca(NO_3)_2$ slugs were irradiated to produce a single curie of ^{14}C; pounds of sulfur were required to make a curie of ^{32}P; ^{60}Co sources were not nearly intense enough. We were helped in this matter quite early when a program was started with Hanford Atomic Products Operation late in 1948 to irradiate targets in their reactors, always taking into account, of course, the necessary security precautions. We were elated with the results of these irradiations and put everything we could into those

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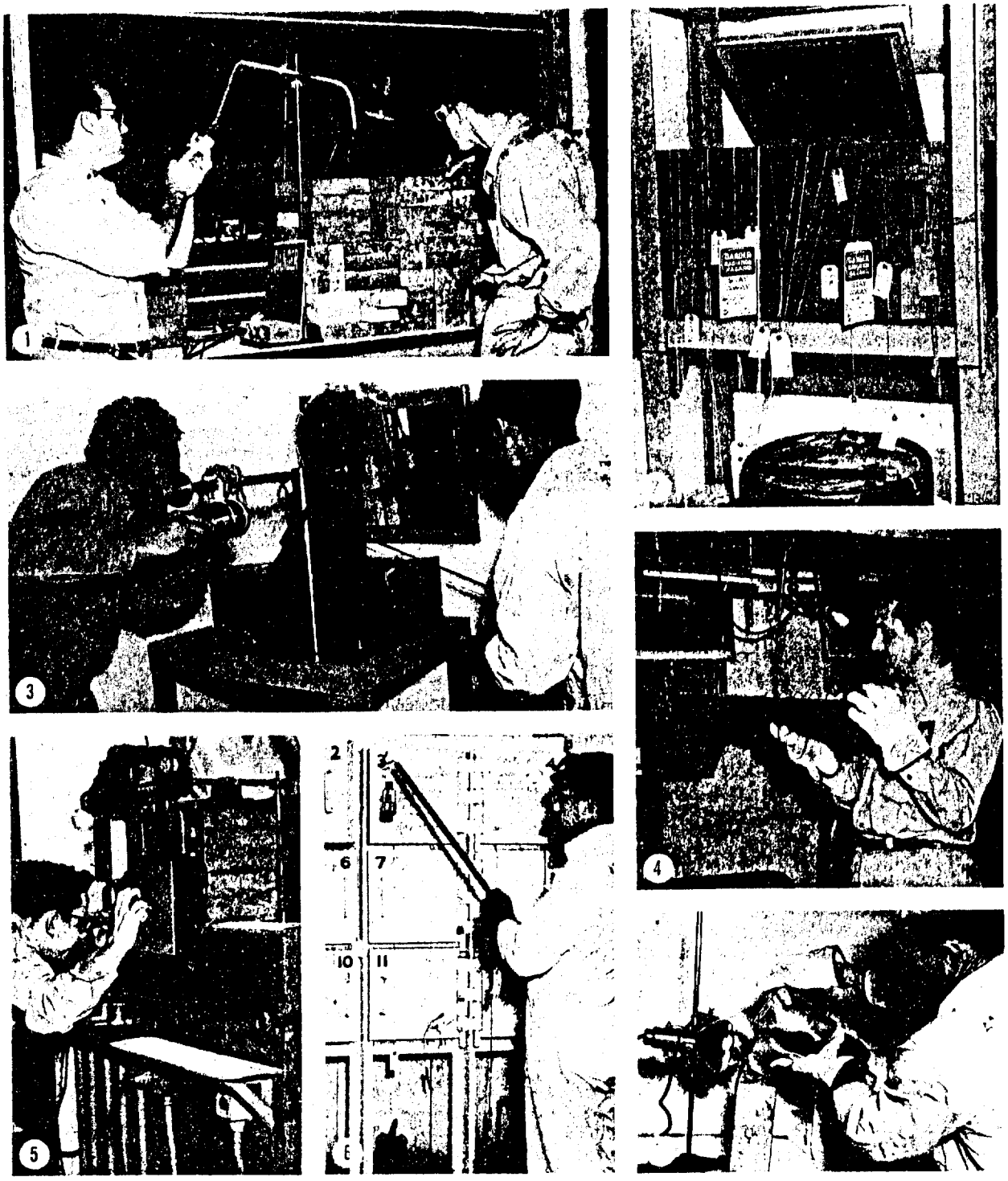

Fig. 3 Milestones and scenes of early radioisotope activities at Oak Ridge. **1.** *Radioisotope Development Laboratory.* **2.** *Storage barricade for radioisotopes awaiting shipment.* **3.** *Radioisotope dispensing equipment, 1947.* **4.** *Irradiation units in graphite stringers going into pile.* **5.** *A hot laboratory in 1946.* **6.** *Storage and handling of radioisotope analytical samples.* **7.** *Loading a sulfur can for Graphite Reactor to produce* ^{32}P. **8.** *Iodine-131 process equipment (from tellurium).* **9.** *Loading the thousandth shipment of radioisotopes, July 9, 1947, to the National Institutes of Health.* **10.** *Early cell for fission-product separations. Element 62 (Pm) was first isolated here.* **11.** *The "Argonne" manipulator cell.* **12.** *Equipment for purification of* ^{131}I *in fission-product iodine plant.* **13.** *Ground breaking for Isotope Area, 1948. Graphite Reactor building is in the background.* **14.** *Shipping containers for irraidation units and processed radioisotopes, 1948.*

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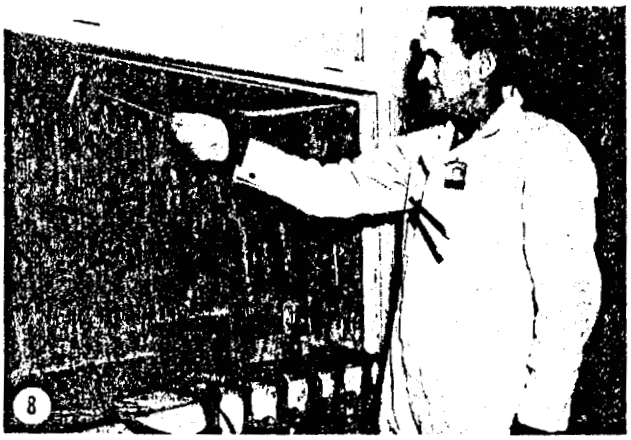

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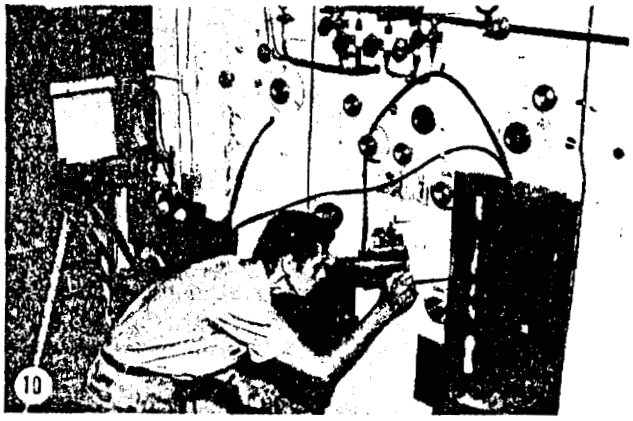

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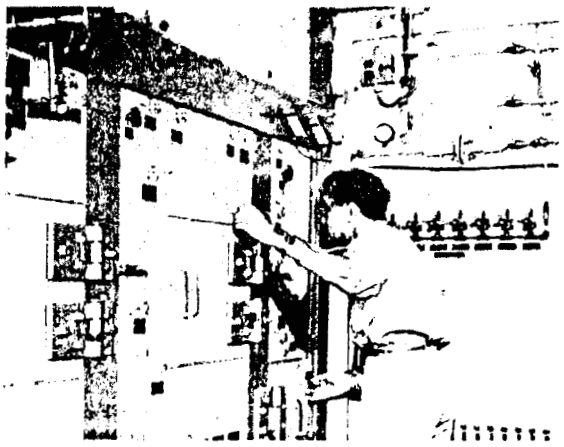

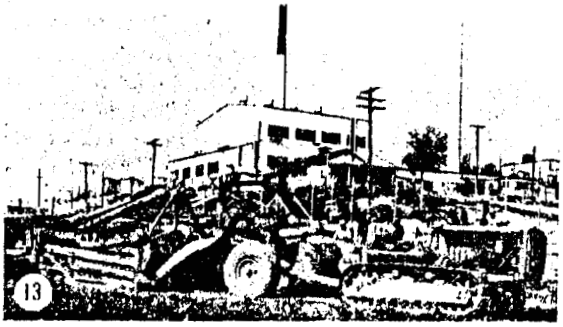

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enormous reactors as soon as possible (helped greatly by the magnificent cooperation of the Hanford technical and operating groups). Of special note was the placing in the reactor of a large charge of our newly developed ^{14}C target material, Be_3N_2, and of many shapes and sizes of cobalt metal. The Hanford reactors continue as mainstays of the radioisotopes production program to this day, being used in later years for target irradiations requiring a large flux volume. The Low Intensity Test Reactor (LITR), the progenitor of the Materials Testing Reactor (MTR), built at ORNL in 1951 primarily for hydraulic-test purposes, was converted into a 1500-kw operating unit and gave us our first high flux [$\sim 3 \times 10^{13}$ n/(cm^2)(sec)] locally. Pneumatic tubes in the LITR also helped greatly in preparing short-lived radioisotopes on fast, convenient schedules. During the first decade these three reactors enabled us to supply the bulk of the radioisotopes used throughout the world. Near the end of this period the MTR began operating, and we at last began to irradiate targets (1955) in a flux that we had only dreamed about not too many years earlier, about 2×10^{14} n/(cm^2)(sec). MTR irradiations, with those following in the Oak Ridge Research Reactor (ORR) at the Laboratory, marked the beginning of a new era in the radioisotope program.

No report would be complete without mentioning the program, even though short in years, of providing ^{14}C-labeled compounds. To make more materials available for research, the U. S. Atomic Energy Commission (AEC) distributed in the late 1940's and early 1950's ^{14}C-labeled compounds that were produced by the ORNL Chemistry Division. With the advent of commercial availability of these products, the AEC discontinued this work. This phase of the program ended with a "going-out-of-business sale" of the labeled compounds that were in inventory in 1955.

Highlights of Early Reports

On the basis of a quick review of reports of the "early days" (1946–1956), we find it difficult to single out any "most significant" points since, in those days, nearly everything accomplished appeared significant. However, we have chosen to mention a few highlights, either as anecdotes or as major accomplishments.

CYCLOTRON ISOTOPES

It is interesting that we were receiving targets from the cyclotrons at the Massachusetts Institute of Technology, University of Pittsburgh, University of California at Berkeley, and Washington University at St. Louis as early as 1948 for processing into high-quality solution-form preparations of ^{22}Na, ^{59}Fe, ^{73}As, etc. In this way radioisotopes that could not be easily produced by neutron reactions were made available to many scientists throughout the world. In 1953 we began to use the powerful ORNL 86-in. proton cyclotron for irradiating targets from which processed isotopes were prepared; later, processing service was discontinued by the AEC, and today only the unprocessed, irradiated targets from this machine are sent out.

MODERN HOT CELL

In 1952 we installed one of the first modern hot cells, complete with manipulators and viewing windows, which we dubbed the "Argonne" cell in honor of the workers at ANL who had done such a marvelous job in developing remote-control devices. The original window contained zinc bromide solution, which was subsequently replaced by a high-density glass to more nearly provide the shielding equivalent of the high-density concrete walls of the cell. The Argonne cell is still in daily service.

FISSION-PRODUCT ^{137}Cs

One of the interesting sidelights is the development of the ^{137}Cs (fission product) program. Late in 1948 the National Bureau of Standards (NBS) wanted a 10-curie source of ^{137}Cs. We had been interested in ^{137}Cs earlier but had only time to do some literature and laboratory work. The NBS source was prepared by setting up a cesium alum process in a 50-gal tank on a pad, shielded by concrete blocks, in the ORNL Tank Farm Area. The original equipment was so crude that the agitator in the tank was hardly more than a remote-control hand-operated paddle. The completed 10-curie source was delivered to the NBS in 1950. From this beginning we improved the equipment's scale and versatility, and in 1954 we reached the significant milestone of producing a 1500-curie teletherapy source for use at the Oak Ridge Institute of Nuclear Studies (ORINS) Medical Division.

LARGE AND INTENSE SOURCES

Radiographers and medical radiation therapists were interested very early in obtaining intense sources of ^{60}Co, ^{137}Cs, and ^{192}Ir to supplement radium sources and X-ray machines. Radiologists also needed large sources for teletherapy in treating deep tumors. We developed techniques for preparing a variety of ^{60}Co sources, ranging from tiny wires (to replace radon seeds) to 1000-curie sources for teletherapy. About the same time, researchers on radiation effects wanted large sources for irradiators. Since the radiographer commonly used "inches" when describing sources, whereas the teletherapist was more familiar with "centimeters," "building blocks" were made to satisfy all potential users: standard-sized pieces of cobalt were irradiated, including $1/16$- by $1/16$-in. to $1/8$- by $1/8$-in. right cylinders, 1- by 1-cm and 2-mm by 1-cm disks, and 2-cm by

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1-mm wafers. The production of ^{60}Co was divided between Hanford and the MTR. Hanford reactors provided cobalt for radiography, and the MTR, material for teletherapy. The ever-increasing requirement for higher specific activity and the growing total demand for teletherapy material prompted us in 1956 to ask the AEC's Savannah River Plant to produce ^{60}Co for teletherapy sources; when these reactors were used, the logjam soon was broken. An interesting sidelight was the evolution of sealing techniques for encapsulated sources. The technology proceeded from single to double encapsulations with lead gaskets and screw threads and then from soldering to Heliarc welding. This evolution was intimately tied to improvement in remote-handling techniques in manipulator cells.

COBALT-60 STORAGE "GARDEN"

As a by-product of the ^{60}Co program, we completed in 1956 a storage facility for ready accessibility and dry storage of cobalt. Included in the design was a cavity for using the radiation emanating from the 100 thousand to 300 thousand curies of cobalt that were kept in inventory. The facility was named the Cobalt Storage Garden and Irradiator. The irradiator cavity provided 1 cu ft of dry irradiation space with a gamma dose rate in excess of 1×10^6 r/hr (the maximum dosage was dependent upon the configuration used in loading the storage tubes surrounding the chamber). Many materials have been irradiated in this cavity, but one we will not forget was the perfume brought to the Laboratory by a perfume expert. After irradiation at various dosages, the results, good or bad, were determined by the expert's smelling the samples.

FISSION PRODUCTS

Fission products were always a special category of radioisotopes—not "waste," but rather a bountiful supply of inexpensive radioactive and stable isotopes. Further, many of the nuclides that occur in fission are difficult to make in high specific activity by other neutron reactions. The short-lived fission products processed in the early days were ^{131}I, $^{95}Zr-^{95}Nb$, ^{140}Ba, ^{89}Sr, ^{141}Ce, ^{103}Ru, ^{91}Y, ^{147}Nd, and later ^{133}Xe. The feed for early work on these radioisotopes came from the effluent of an early classified program. Later, when this source of supply was no longer available, the irradiation of tellurium was considered for the specific production of the important isotope, ^{131}I. However, when it became apparent that meeting the demand for ^{131}I would require an inordinate amount of space in the Graphite Reactor, the decision was made to produce fission-product iodine by processing uranium "slugs." Now, from enriched-uranium targets irradiated in the ORR, this is still a source of high-specific-activity ^{131}I and other fission-product radioisotopes. The major effort and accomplishment in this program was the development of processes and relatively small-scale process units for separating and purifying these shorter lived materials. The work on longer lived fission products was concentrated on ^{137}Cs, ^{90}Sr, ^{106}Ru, ^{147}Pm, ^{144}Ce, and ^{99}Tc. In the early days a variety of processes were tried, including ion exchange. We remember well our initiation to the gassing problem when 1 curie of ^{90}Sr was placed upon a small column (~1 cm in diameter by 25 cm long). None of the solution added to the head tank flowed through the column because of the back pressure developed by gases formed through radiolysis! However, the development of appropriate processes led eventually to the large-scale separation and purification of these long-lived species. Some of the most difficult and exasperating experiences related to the procurement of appropriate feed materials, which were supplied from the ORNL Tank Farm, Idaho Chemical Processing Plant, and ultimately Hanford. The work was especially satisfying, however, because it culminated in the design and construction of the Fission Products Pilot Plant during 1954–1956. Few people believed that fission products would amount to anything, and appropriations for radioisotope capital equipment were always hard to get. Nevertheless, with the help of many people at various levels of company and government management, F3P (as it came to be called) was completed and operated with signal success, furnishing a fitting climax to the first decade of the radioisotope program.

In retrospect, these were probably the most exciting days of our lives. Even though many of them were trying, we always felt a sense of challenge and accomplishment while learning, doing, and making these interesting and useful materials available for use in such a variety of fields.

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