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RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR AUGUST 1971

A. F. Rupp

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Isotopes Development Center

RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR AUGUST 1971

A. F. Rupp

Work Sponsored by

AEC Division of Isotopes Development

SEPTEMBER 1971

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RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR AUGUST 1971

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RADIOISOTOPE PRODUCTION AND MATERIALS DEVELOPMENT

REACTOR-PRODUCED ISOTOPES - 08-01-01

A. Biomedical Radioisotopes

1. Phosphorus-33

The purpose of this project is to develop methods of preparing hundred-millicurie quantities of carrier-free phosphorus-33 containing $\leq 5\%$ phosphorus-32. The two methods of producing phosphorus-33 being evaluated are based on the irradiation of highly enriched targets of sulfur-33 (>92 at. %) or chlorine-36 (approximately 63 at. %) in a fast neutron flux. Phosphorus-33 (25.2 days; 0.248-MeV β^-_{max}) has both a longer half-life and a lower energy beta than phosphorus-32 (14.3 days; 1.709-MeV β^-_{max}), which makes it advantageous for autoradiography, longer ecological and agricultural experiments than with phosphorus-32, synthesis of tagged complex organo-phosphorus compounds, and double labeling experiments.

Target material from two earlier targets which produced a phosphorus-33 product with 13% phosphorus-32 was reprocessed to remove sulfate by precipitation of the barium salt; 2.13 g of $K^{36}Cl$ with an isotopic assay of 69% chlorine-36 was recovered from targets originally thought to contain 2.78 g.

The recovered material was equally divided, and two reactor targets were fabricated. One was used to replace a $K_2{}^{33}SO_4$ target which was removed after a three-cycle irradiation. The $K_2{}^{33}SO_4$ target is expected to yield about 0.5 Ci of phosphorus-33. The product which was 13% phosphorus-32 will have decayed to about 150 mCi of phosphorus-33 with phosphorus-32 under 5% by September.

2. Potassium-43

The objectives of this project are: to prepare potassium-43 by the $^{43}Ca(n,p)^{43}K$ reaction, using isotopically enriched ^{43}CaO targets, in quantities sufficient for medical and biological experiments; to define a method for separating potassium-43 from the target in a purity suitable for medical use; and to establish cooperative programs with medical institutions interested in evaluating its usefulness. Potassium-43, with a half-life of 22.5 hr and gamma-ray

emissions of 0.373 and 0.617 MeV, is potentially useful for metabolic and clinical studies of blood flow, rejection of transplanted organs, and kidney function because the dose rate would permit multiple doses.

Two batches of potassium-43 were made. Data for the runs are summarized in Tables 1 and 2.

Shipments were made under our medical cooperative programs to Johns Hopkins Medical Institution, Peter Bent Brigham Hospital, University of Mississippi Medical Center, and University Hospitals of Cleveland.

Table 1. Yield of Potassium-43 from Calcium-43 Targets

	Target 398	Target 385
Weight ^{43}CaO (mg)	125[a]	122[b]
Irradiation time (hr)	61.8	65
Product delivery date	7/27/71	8/10/71
Total product at 8:00 AM on delivery date (mCi)		
Potassium-43	6.5	7.1
Potassium-42	0.7 (11.5%)	0.8 (11.9%)

[a]61% calcium-43 recovered from target 382.
[b]Approximately 50% calcium-43 recovered in July 1971.

Table 2. Radionuclides Found in Dissolver Sample by Gamma Scanning

Radionuclide	Half-Life	Activity at Discharge[a] (mCi)		Activity Concentration at Discharge (μCi per mg of target)	
		Target 398	Target 385	Target 398	Target 385
Potassium-43	22.5 h	16.5	16.7	132	137
Potassium-42	12.4 h	3.9	4.5	31	37
Sodium-24	15.0 h	3.5	1.8	28	15
Manganese-56	2.58 h	8.5	9.7	67	79
Strontium-87m	2.83 h	30.6	6.6	240	54
Calcium-47	4.53 d	0.26	0.35	2.1	2.9
Scandium-46	83.8 d	0.6	4.8	4.6	39
Scandium-47	3.35 d	0.14	0.32	1	3
Samarium-153	46.8 h	0.09	—	0.7	—
Copper-64	12.8 h	1.9	1.4	15	11
Lanthanum-140	40.2 h	0.05	—	0.4	—
Cadmium-115	53.5 h	0.03	—	0.2	—
Chromium-57	27.7 d	0.06	—	0.5	—
Gold-199	3.15 d	0.06	—	0.5	—
Bromine-82	35.5 h		0.1		0.6

[a]Total target.

B. Isotopic Power

1. Thulium-170

The objectives of this research are to provide sufficient data on thulium-170 to permit assessment of its potential application as an isotopic power source for short-duration missions.

The experimental runs to determine the vapor pressure of Yb_2O_3 have been made at 1990°C using the large Knudsen cell. The results shown in Table 3 have been computed based on the assumption that Yb_2O_3 vapor at 1990°C dissociates into ytterbium and oxygen. This assumption is supported by the published mass spectrometric data. The "apparent" vapor pressure of Yb_2O_3 at 1990°C is 2.22×10^{-6} atm which is approximately nine times that of Tm_2O_3 at the same temperature (the value obtained with the large cell).

Table 3. Rates of Effusion, and Partial and Total Pressures of Various Vapor Species Above Solid Yb_2O_3 in Large Knudsen Cell at 1990°C

Vapor Species	Rate of Effusion[a] ($g/cm^2.min$) x 10^4	Partial Pressure[b] (atm) x 10^7
Yb	11.05	15.24
O	1.53	6.95
Total	12.58	22.19[c]

[a]Corrected for the weight change of the empty Knudsen cell as well as for the thermal expansion of the orifice.
[b]Corrected for the Clausing factor (a transmission probability).
[c]"Apparent" vapor pressure of Yb_2O_3.

Some deposits around the orifice were visible under the microscope (approximately 100X) after a few runs, but most of the deposited material was removed by heating the empty cell at 10^{-6} torr before the vapor pressure run was resumed. Although the residual dark crystalline material around the orifice has not yet been identified, it could be some Yb-W intermetallic compound and/or YbO.

In progress is the determination of the rate of weight change of the empty Knudsen cell at 1900°C and 10^{-6} torr to be used in the correction of the vapor pressure data.

C. Reactor Products Pilot Production (Production and Inventory Accounts)

Processed Units		Service Irradiations	
Radioisotope	Amount (mCi)	Type	Number
Calcium-47	11	Platinum-196	1
Copper-67	23		
Nickel-63	14,500		
Silver-111	339		

D. Miscellaneous

1. HFIR Target Testing

Two hydraulic tube rabbits were tested this month with no failures.

ACCELERATOR-PRODUCED ISOTOPES - 08-01-02

A. Biomedical Radioisotopes

1. Gallium-67

The objectives of this program are to determine the optimal target configuration for gallium-67 (78.2 hr) production by the $^{68}Zn(p,2n)^{67}Ga$ reaction in acceptable purity and quantity and to provide gallium-67 for clinical applications research and development. Interest in this isotope has been spurred by evidence, obtained by the Medical Division of Oak Ridge Associated Universities (ORAU), of a high uptake of carrier-free gallium-67 by lymphoid tumors in both animals and humans.

Gallium-67 decays by electron capture with the emission of four main gamma rays of 93, 185, 300, and 394 keV with intensities of 42, 24, 17, and 5%, respectively.

Weekly pilot production of gallium-67 was continued; four preparations were shipped to customers. Four weekly shipments of 100-mCi batches of gallium-67 citrate were supplied to ORAU during this period as part of the Cooperative Group to Study Localization of Radiopharmaceuticals (CGSLR) project. One positive pyrogen test was reported for one of these products. Additional testing of this sample, i.e., material from the same bottle, and testing of other bottles of this preparation indicated that the product was apyrogenic. ORAU concluded that the positive test results should be attributed to either the rabbits, which had not been preconditioned, or contamination of the solutions used to dilute the preparation for testing, rather than to contamination of the product as prepared at ORNL. This 100-mCi batch was not shipped by ORAU to participants of the CGSLR for use. It is felt that since the positive pyrogen test was not attributed to the ORNL product no change in operating procedures is required.

2. Indium-111

The objectives of this program have been to define and optimize the pertinent production parameters for the economical production of indium-111 of satisfactory quality for radiopharmaceutical manufacture. The current objectives are to obtain practical operating experience of the procedures developed by pilot production and to provide this radionuclide to interested customers as a special research material — in batch lots — for their evaluation. Indium-111 has been suggested for such studies as spinal-cerebral cisternography, aerosol lung studies, delayed brain scanning, visualization of the lymphatic system, metabolic studies of indium-labeled macroaggregates and colloids, and tumor localization. Indium-111 has gamma emissions of 173 keV (89%) and 247 keV (94%) ideally suited for external detection and an optimal half-life (2.81 days) for labeling and distribution studies which must be carried out over 24 hr or longer.

One batch of indium-111, approximately 120 mCi total at end of one-day decay allowance, was prepared and portions were supplied to interested clincians as a special research material. Interest in indium-111 has developed to the point that the pilot production schedule will be increased to every-other-week production until mid-September, whereupon weekly production will be undertaken for a limited trial period dependent upon continued demand.

An article entitled "Cyclotron Production of Carrier-Free Indium-111" has been accepted for publication in the *Int. J. Appl. Radiat. Isotopes*. This article discusses our accumulated indium-111 production and processing experience.

B. Accelerator Pilot Production (Production and Inventory Accounts)

Table 4 gives the August 1971 accelerator runs for ORNL and non-ORNL programs.

Table 4. Accelerator Irradiations and Runs for August 1971

Product	No. of Runs	Time (hr:min)			Total Charges
		Beam	Misc.	Total	
		ORNL Programs			
Cobalt-61	6	18:15	9:10	27:25	$2,672
Gallium-67	5	20:50	5:35	26:25	2,696
Cadmium-109	1	7:00	1:15	8:15	1,093
Indium-111	1	2:30	1:10	3:40	380
Total		48:35	17:10	65:45	$6,841

Table 4. Continued

Product	No. of Runs	Time (hr:min) Beam	Misc.	Total	Total Charges
		Non-ORNL Programs			
Gold-195	1	8:00	1:15	9:15	$1,867
Rubidium-84	1	5:35	1:15	6:50	1,588
Tantalum-179	1	1:45	1:15	3:00	439
Total		15:20	3:45	19:05	$3,894

FISSION PRODUCTS - 08-01-03

A. Krypton-85 Enrichment

One standard liter of krypton gas containing 43.8% krypton-85 was removed from unit CD (see Fig. 1). The count rate at the center section of this unit reached a maximum of 19,000 counts/min, but a plant power outage left the thermal diffusion units down long enough to drop the count rate to 16,950 counts/min. The product was removed at this lower count rate. The power failure also caused a count rate drop due to back diffusion and remixing on units C and D.

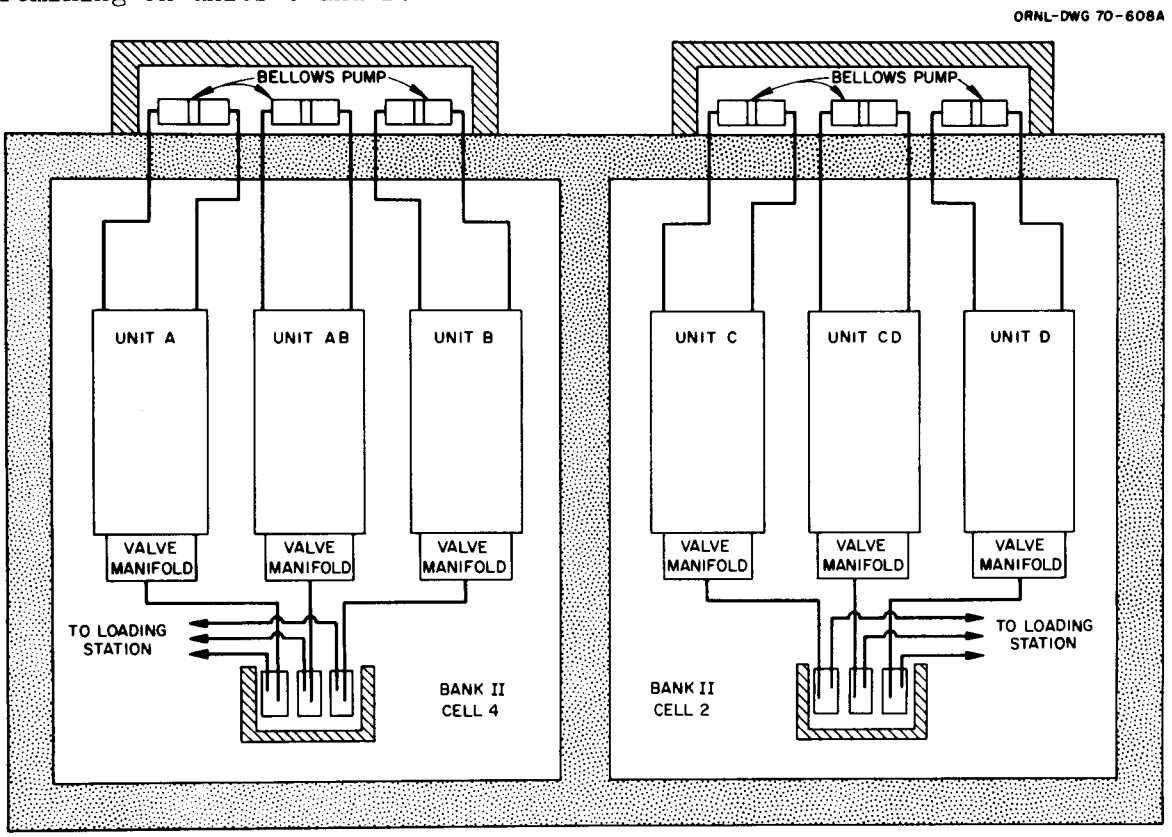

Fig. 1. Schematic Arrangement of Krypton-85 Columns.

Unit AB has been loaded with fission xenon in an experiment to determine the effectiveness of thermal diffusion in depleting the trace of fission krypton found in the xenon.

Unit	Activity in Unit (Ci)	Time Since Last Product Removal (days)	Count Rate in Product Section (counts/min) July 1971	Aug. 1971
A	120	40	2,250	2,250
AB	Xenon	—	—	—
B	120	40	2,300	2,400
C	1784	230	9,500	7,200
CD	2056	480	18,700	11,700
D	1647	230	9,200	7,200

B. Cesium-137 Gamma Source Development

Cesium-137 chloride has been the compound of choice for radiation sources in applications of moderate temperature conditions. In many respects cesium chloride is the ideal compound; the cesium weight per unit volume is high, the radiation resistance of the CsCl is excellent, the compatibility with stainless steel at ambient temperatures is excellent, and its preparation is straightforward. However, the projected conditions of use of cesium-137 gamma sources are increasingly severe with respect to temperature, and some applications indicate the need for a cesium source material which has low solubility. In view of these projected conditions of use, the testing of $^{137}CsCl$ at elevated temperatures is being done and the development of a low solubility, temperature-resistant source form of cesium is being studied.

1. Alternative Cesium-137 Source Form Development

a. Methods of Preparation

A stagewise reaction method was employed in the preparation of compounds $Cs_2O \cdot MgO \cdot nSiO_2$ and $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$. The method consists of first reacting Cs_2CO_3 with SiO_2 (colloidal) to form the water-glass-like compound $Cs_2O \cdot nSiO_2$ by

$$Cs_2CO_3 + nSiO_2 \rightarrow Cs_2O \cdot nSiO_2 + CO_2\uparrow \qquad (1)$$

The compound $Cs_2O \cdot nSiO_2$ thus formed was then reacted with MgO and Al_2O_3, separately, to produce $Cs_2O \cdot MgO \cdot nSiO_2$ and $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$. In both reaction steps, the slurry reaction was followed by calcination at high temperatures. The calcining conditions were determined based on the behavior of the reaction mixture in the high-temperature stage of the optical microscope.

b. Characterization of $Cs_2O \cdot nSiO_2$ Products

Summarized in Table 5 are the chemical composition (calculated in terms of atomic ratio Si/Cs) and stability in water of the products from the first reaction step [see Eq. (1)]. Since $Cs_2O \cdot nSiO_2$ is expected to be water soluble, the amount of insolubles in the product can be used as a guideline in estimating the extent of the reaction. Table 5 demonstrates that the conversion in the product from the slurry reaction at up to 165°C (Samples AU-1, -4, and -7) is low, as a considerable amount of insolubles (30-53%) is still present. As can be seen from the very low contents (0.7-0.8%) of insolubles for samples AU-2, -3, -5, -6, -8, and -9, calcination of the product at 1000°C is sufficient to render the reaction practically complete. A comparison of samples AU-2, -5, and -8 with AU-3, -6, and -9 reveals that an extended calcining time is unnecessary to achieve a high conversion.

Table 5. Characteristics of Compounds $Cs_2O \cdot nSiO_2$

Sample No.[a]	Drying or Calcining Temperature (°C)	Atomic Ratio Si/Cs		Wt % Insolubles in Distilled Water
		Feed	Product	
AU-1	165	0.499	0.69	33.1
AU-2	1000	0.499	0.65	0.85
AU-3	1000	0.499	0.77	0.79
AU-4	165	0.998	1.18	29.9
AU-5	1000	0.998	1.15	0.77
AU-6	1000	0.998	1.11	0.71
AU-7	165	1.997	1.83	53.3
AU-8	1000	1.997	1.92	0.72
AU-9	1000	1.997	1.92	0.85

[a]The calcining time for AU-3, -6, and -9 was three times as long as AU-2, -5, and -8.

There appears to be some trend in the variation of the Si/Cs ratio of the product with that of the feed. An increase in the product Si/Cs ratio as compared with the feed Si/Cs ratio presumably implies a preferential removal of cesium oxides. The relative increase in the Si/Cs ratio is especially notable when the ratio in the original feed is low (i.e., 0.499). With a high Si/Cs ratio in the feed (1.997), there is a slight drop in the ratio for the product.

c. Characterization of $Cs_2O \cdot MgO \cdot nSiO_2$ and $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$

Table 6 shows some properties of the products from the second reaction step. In this case, the amount of insolubles may be considered as an indication of the extent of conversion to the desired compound. Comparing the weight percent insolubles for $Cs_2O \cdot MgO \cdot nSiO_2$ products with the MgO content of the initial reaction mixture (9.1 wt % which is essentially water insoluble), it is seen that there was an appreciable degree of conversion at the conclusion of the slurry reaction (20.8% insolubles, sample AU-10). Calcination of the product at 1100°C increased the conversion (49.3% insolubles, AU-11), but an extended calcination time at this temperature did not seem to improve the conversion any further (44.2% insolubles, AU-12). Similar behavior is observed for the $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$ products, but the degree of conversion in the slurry reaction step (22.9% insolubles, AU-13) was probably small since the Al_2O_3 content (water insoluble) of the initial mixture was 20.3 wt %.

Table 6. Characteristics of $Cs_2O \cdot MgO \cdot nSiO_2$ and $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$ Products

Sample No.[a]	Drying or Calcining Temperature (°C)	Atomic Ratio[b] Si/Cs	Mg/Cs	Al/Cs	Wt % Insoluble in Distilled Water[c]
		$Cs_2O \cdot MgO \cdot nSiO_2$			
AU-10	140	1.03	0.54	—	20.8
AU-11	1100	1.08	0.54	—	49.3
AU-12	1100				44.2
		$Cs_2O \cdot Al_2O_3 \cdot nSiO_2$			
AU-13	140	0.68	—	1.03	22.9
AU-14	1100	0.84	—	1.23	40.2
AU-15	1100				37.7

[a]The calcining time for AU-12 and AU-15 was four times as long as that for AU-11 and AU-14.
[b]The Si/Cs, Mg/Cs, and Al/Cs ratios of the original mixtures were 1.25, 0.54, and 1.08, respectively.
[c]The MgO content of the initial reaction mixture of $Cs_2O \cdot MgO \cdot nSiO_2$ was 9.1 wt % while the Al_2O_3 content of $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$ was 20.3 wt %. They are practically water insoluble.

In regard to the atomic ratio of various elements, Table 6 indicates that the shift in the Mg/Cs and Al/Cs ratios as compared with the initial mixture was rather small. However, a significant reduction in the Si/Cs ratio is noted (approximately 20-50% drop) for all the products, presumably indicating preferential losses of some silicon compound. Calcination at 1100°C tends to increase the Si/Cs and Al/Cs ratios. The chemical analysis of silicon is being reexamined to confirm the variation in the Si/Cs ratio discussed above.

d. Assessment of Available Results

Compounds of the types $Cs_2O \cdot MgO \cdot nSiO_2$ and $Cs_2O \cdot Al_2O_3 \cdot nSiO_2$ can be prepared by first forming the water-glass-like compound $Cs_2O \cdot nSiO_2$, followed by reaction with MgO and Al_2O_3, respectively, and finally calcining the products at 1100°C. The products thus obtained would contain 40-50 wt % of insoluble compounds. However, further increase in the amount of insolubles (and therefore higher degrees of conversion to the desired compounds) can be expected by a slight modification of the preparation method used, e.g., by increasing the calcining temperature above 1100°C.

C. Cesium-137 Pilot Production (Production and Inventory Accounts)

1. Processing and Process Status

Six batches of $^{137}CsCl$ were prepared from the cesium carbonate processed during July; this material was from the first HAPO-I-C shipment. Analysis of the products is not complete, but preliminary analysis indicates the total yield was 70,000 Ci of good quality product. Elemental analysis of representative samples showed impurities totaling less than 0.5 wt %, with the major impurity being rubidium at 0.09 wt %. All recycled cesium-137 (from oxalate filtrates, equipment washes, etc.) was accumulated in the crystallizer system; then the system was secured for the unloading of the second HAPO-I-C cask.

The HAPO-I-C cask was received on August 19 and inserted into the unloading cell. The quoted loading on this cask is 380,000 Ci; it will be unloaded in September. The current cesium-137 process status is as follows:

Item	Cesium-137 (Ci)
In-process material	964,000
$^{137}CsCl$ product	47,500
Sources in fabrication	30,500
Completed sources awaiting shipment	46,500

2. Operational Summary

	August 1971		FY 1972	
Item	No.	Amount (Ci)	No.	Amount (Ci)
HAPO shipments received	1	380,000	1	380,000
Product batches prepared	6	70,000	6	70,000
Sources fabricated	15	18,500	15	18,500
Special form containers loaded	13	600	13	600
Sources shipped	4	11,500	4	11,500
Special form containers shipped	11	500	11	500

3. Current Orders

Current orders for cesium-137 as sources or bulk powder are shown below:

Customer	Amount (Ci)	Estimated Shipping Date
J. L. Shephard and Assciates	7,050	September 1971
Lockheed-Georgia Company[a]	∿35,000	September 1971
American Nuclear Corporation	125	September 1971
Brookhaven National Laboratory	203,000	FY 1972
Total	245,175	

[a]The cesium-137 activity to be supplied by Lockheed-Georgia Company.

An order is on hand for approximately 154,300 Ci as bulk powder, to be scheduled and shipped as released by the customer.

4. Source Fabrication

The cesium-137 campaign to make a variety of sources is still in progress. The following orders have been filled.

Customer	Number of Sources	Type	Curies per Source	Total Curies
Mexico (government)	2	Radiographic	7	14
AECL	1	Teletherapy	1,500	1,500
USAEC	1	Tubular	10,000	10,000
J. L. Shephard & Assoc.	11	Miscellaneous	Varied[a]	6,950
Total	15			18,464

[a]Between 10 and 6000 Ci/source.

USAEC Order - A cesium-137 irradiator, which originally contained 9900 Ci of cesium-137 when it was fabricated in 1964, was returned to ORNL from the Puerto Rico Nuclear Center to be defueled and a fresh source installed. The source had been immersed in oil which had solidified causing some difficulty in the defueling operation. A new source, containing 10,000 Ci cesium-137, was fabricated and loaded into the irradiator. The irradiator was then shipped to the USDA, North Central Forest Experiment Station in Rhinelander, Wisconsin.

J. L. Shephard and Associates Order - Eleven cesium sources of various sizes and curie content were fabricated and delivered to the Shipping Department. Shipment of these sources is being delayed until the necessary fire shield is available. The carrier furnished by the customer was inadequate thus necessitating the use of an ORNL carrier.

The following powder cans were loaded with cesium carbonate and shipped:

Customer	Number of Cans	Curies/Can	Total Curies
U. K. Treasury & Supply Deleg.	10	50	500
American Hoechst Corporation	1	5	5

In addition, two powder cans containing 50 Ci each of cesium carbonate were made for stock.

An order for sixteen cesium-137 sources, each containing approximately 1900 Ci, for the Lockheed-Georgia Company is now in progress and should be completed in August.

One 125-Ci cesium-137 source for American Nuclear Corporation is also being fabricated.

D. Strontium-90 Pilot Production (Production and Inventory Accounts)

1. Processing and Process Status

Removal of the large pieces of out-of-service equipment from the strontium-90 pressing cell was accomplished with some difficulty, and decontamination of the cell for new equipment insertion continued. The strontium-90 processing system should be ready to resume operation by the first week in September.

Due to the unusually high content of inert rare earths in the most recently received strontium-90 feed (rare earth/strontium weight ratio 0.07), two 500-Ci scale test runs were made on this feed to investigate possible methods for removal of rare earths. It was found that the normal process for strontium-90 titanate preparation (precipitation by carbonate in presence of TiO_2) does not result in any significant decontamination from rare earths. The rare earths can be separated by precipitation as the oxalates from 0.1 *M* HNO_3 (decontamination factor with respect to strontium of >20) with low strontium-90 losses. This procedure, however, results in a strontium-90 feed containing oxalate and this may influence subsequent processing. Tests are being made to determine the optimum conditions for good rare-earth removal, low strontium-90 loss, and minimum oxalate concentration in the filtrate. The next large strontium-90 project will require approximately 325,000 Ci of strontium-90 in the distrontium titanate form at an effective power density of 1.2 W/cm^3. Without separation of the rare earths from the feed, this power density would be difficult to achieve.

Two SNAP-21 sources containing a total of 49,900 Ci of strontium-90 were returned from the U. S. Navy for storage at ORNL. The current strontium-90 process status is as follows:

Item	Strontium-90 (Ci)
In-process material	959,000
Strontium-90 products	439,500
Sources in fabrication	0
Returned SNAP sources	340,900
Completed sources awaiting shipment	64,600

2. Operational Summary

Item	August 1971		FY 1972	
	No.	Amount (Ci)	No.	Amount (Ci)
HAPO shipment received	0	0	0	0
Product batches prepared	0	0	0	0
Sources fabricated	0	0	0	0
Special form containers loaded	0	0	0	0
Sources shipped	0	0	0	0
Special form containers shipped	0	0	0	0
Shipments to customers	0	0	12	45,900

3. Current Orders

Current orders for strontium-90 as sources or bulk powder are as follows:

Customer	Amount (Ci)	Estimated Shipping Date
U. S. Navy	411,380	a
U. S. Navy	208,000	a
Total	619,380	

[a]Further work and subsequent shipment are pending receipt of instructions from customer.

E. Strontium Pellet Vacuum Hot Press

1. Pellet Pressing

Two pellets were pressed during August to examine the effects of rare-earth impurities (simulating current material which contains about 7% of the strontium weight as rare earths) on the pressing characteristics of Sr_2TiO_4. The Sr_2TiO_4 was prepared by coprecipitating $SrCO_3$ and TiO_2 and calcining at 1300°C for 16 hr.

Both pellets were pressed in a TZM die body with a graphite liner to prevent interaction between the Sr_2TiO_4 and the TZM.

Pellet 19 - This pellet was pressed at 4500 psi between 1360 and 1400°C. No screens were used to reduce the effects of thermal shock and as a result the pellet was extensively cracked. The density of the pellet was 4.3 g/cm^3.

Pellet 20 - This pellet was made from a second batch of powder identical to that used in pellet 19 except no praseodymium was used. Two nickel screens were used to reduce cracking. The pellet was pressed at 4500 psi at a temperature of about 1390°C. The pellet had a uniform appearance with a density of 4.4 g/cm^3.

Both pellets 19 and 20 were made using a modified die rest to reduce heat losses from the lower portion of the die body. The modification entailed extending the bottom punch through an opening in the die rest to the bottom of the vacuum chamber. The die body was thus floated on the bottom punch and not supported by the die rest. The die rest was only used to support the reflector shield.

No significant difference in the appearance of the pellets was observed as a result of the die rest modification.

2. Dry Mixing of $SrCO_3$ and TiO_2

As an alternative to the coprecipitation method whereby $SrCO_3$ and TiO_2 existing as suspended precipitates in a slurry are filtered and calcined to form Sr_2TiO_4 (or other SrO-TiO_2 compounds depending on mole ratios), a dry blending method is being studied. In this method the strontium is precipitated out as $SrCO_3$, dried, analyzed, and blended with dry TiO_2 in appropriate quantities to form the desired titanate compound after calcining.

a. Vee Blender

The first mixing technique studied was a Vee Blender. The dry $SrCO_3$ was mixed with dry TiO_2 (0.5 mole titanium per mole strontium). The material was blended overnight (21 hr), and a sample was taken. The sample was calcined at 1300°C for 16 hr and analyzed by means of x-ray diffraction. The analysis determined that the material was Sr_2TiO_4 — an indication that the blending time was adequate for sufficient mixing. Other samples were taken at later times up to 42 hr and agreed with the 21-hr sample.

b. Waring Blender

A second mixing technique examined was the Waring Blender. The dry materials were mixed in the same proportions as with the Vee Blender. Samples were taken over 3-min increments for a total of 15 min. After each 3-min run, the material in the blender was hand stirred. The five samples were calcined at 1300°C and are currently being analyzed.

F. Short-Lived Fission Products Pilot Production (Production and Inventory Accounts)

Isotope	Number of Batches	Amount (Ci)
Xenon-133	3	∿1000
Iodine-131	2	81
Niobium-95	1	58
Ruthenium-103	1	20
Yttrium-91	1	23
Total		1182

SOURCE DEVELOPMENT - 08-01-04

A. Radiation Processing (Not Supported by 08 Program; WFO Account)

Pollution of our rivers by waste effluent from paper mill, textile mill, and sewage plants is a serious problem. The treatment of paper mill effluent with radiation to lower the COD and BOD, to destroy the color, and to alter the colloid to provide better filterability has been investigated over the past several years by laboratories throughout the world. Specific beneficial results have been obtained, but the costs are too high for the treatment of large volumes of water involved in municipal waste. At Oak Ridge National Laboratory, the program has been developed around high pressure (1500 psi) radiolytic oxidation of the industrial waste solution directly from the source.

1. Radiolytic Oxidation of Paper Mill Waste Effluent and Dye Effluent

Experiments have been conducted this month on effluents from a pulp plant in cooperation with a container corporation plant in Alabama.

The static radiolytic oxidation of effluent from bleach operations was investigated. Samples of this effluent were irradiated at doses of 10^6 and 5×10^6 R under 1500 psi oxygen in a cobalt-60 gamma irradiator. The results of this irradiation are shown in Table 7.

Table 7. Effect of Radiolytic Oxidation on Bleach Plant Effluent at 1500 psi Oxygen

Irradiation Dose	COD (mg/liter)	Transmission at (%) 800 mμ	500 mμ	350 mμ
No radiation	298	77	40	7
10^6 R	105	97	90	74
5×10^6 R	8	98	94	81

The brown color of the effluent became much lighter with 10^6 R exposure and almost colorless at 5×10^6 R exposure. The COD of the effluent dropped from 298 to 105 mg/liter with 10^6 R exposure and down to 8 mg/liter at 5×10^6 R exposure. Radiolytic oxidation is concluded to be effective with this effluent.

2. Treatment of Bleach Plant Effluent in Dynamic System

Since the static experiments showed that radiolytic oxidation of the bleach plant effluent was feasible, quantities of this effluent were radiolytically oxidized by pumping the effluent through the laboratory-scale dynamic system described earlier.[1] The solution was pumped at various speeds through a coiled pipe (containing 10-14 mesh charcoal) in the cobalt-60 irradiator. After 26 liters of effluent had been put through, it became evident that the charcoal was not effective in adsorbing the waste effluent impurities until they were destroyed by the gamma irradiation. The results of this experiment are shown in Table 8.

Table 8. Dynamic System to Radiolytically Oxidize Bleach Plant Effluent

Pumping Speed (liter/hr)	COD (mg/liter)	Transmission at (%)		
		800 mμ	500 mμ	350 mμ
Control	298	77	40	7
1.25	202	78	53	9
0.75	133	84	58	11
0.50	79	91	73	21

Very little if any color reduction was observed at the 3.5-liter/hr pumping speed. The color reduction became evident at 1.25 liters/hr and much more so at 0.50 liter/hr. The COD was steadily reduced from 298 originally to 79 mg/liter at 0.50-liter/hr flow.

The addition of $FeSO_4$ to the bleach plant effluent was investigated as a means of reducing the COD and enhancing the color removal. Bleach plant effluent with additions of either 20 or 40 mg/liter iron as $FeSO_4$ was pumped through the dynamic system at 1.25 liter/hr under 1500 psi oxygen to evaluate the effect of iron additives on the radiolytic oxidation. The results are shown in Table 9.

The 20-mg/liter iron additive to the bleach plant effluent showed the best color reduction and COD reduction. The addition of 20 mg/liter iron does not enhance the high-pressure radiolytic oxidation of the bleach plant effluent in the dynamic system.

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[1]A. F. Rupp, *Radioisotope Program (8000) Progress Report for July 1971*, ORNL-TM-3526, Oak Ridge National Laboratory.

Table 9. Dynamic System to Radiolytically Oxidize Bleach Plant Effluent Containing 20 or 40 mg/liter Iron

Sample	COD (mg/liter)	Transmission at (%)		
		800 mμ	500 mμ	350 mμ
Control	298	77	40	7
No iron	202	78	53	9
20 mg/liter iron	153	95	86	49
40 mg/liter iron	197	92	80	32

Analyses of the dye effluent solution will be started in September. The breakdown products of the radiolytic oxidation of the dye effluent will be analyzed within the scope of this work. Three dye structures that have been investigated in a static irradiation system will be pumped through the dynamic system. This system is now being altered to get more efficient use of the radiation field so that higher pumping speeds will be possible.

B. Radioisotope Characterization, Quality Control, and Standards

1. Radioisotope Characterization

An inquiry about the half-life of plutonium-241 lead to a review of recent published and unpublished data, with the conclusion that a selection of 14.8 ± 0.3 years would encompass all current values which are plausible. Another inquiry resulted in the calculation of helium and heat outputs of a 20-Ci americium-241 source, 0.9 cm^3/year, and 0.16 cal/sec. Strontium-90-yttrium was suggested for use in a gage for ∿5-mil steel or tin sheets.

2. Radioisotope Special Analysis and Quality Control

A leading commercial supplier of radioisotopes and radioactivity standards has requested permission to reprint the report of the NRC standards panel for distribution to customers. The report has also been cited in *Science Trends*. From mass-spectral data on a sample of irradiated nickel-58, the effective capture cross section of nickel-59 has been estimated to be 85 barns, and the optimum irradiation time for production of nickel-59 was calculated to be about 200 days at a neutron flux of 2×10^{15}. The decay scheme, assay methods, and standards for iodine-125 were reviewed. For tritium in "smears," addition of the paper directly to a scintillator mix was recommended. Following agitation and counting in a liquid scintillation counter, the efficiency for that particular sample would be determined by addition of a known quantity of tritiated toluene (calibrated against a similar NBS standard), and recounting. Analytical methods and recent results were reviewed in connection with raw material for strontium-90

production. The methods are essentially as listed in a report,[2] with the addition of spark source mass spectrometry.[3] In answer to an inquiry about determination of a trace of thorium in purified uranium, the following plan was suggested. Thorium can be separated by lanthanum fluoride carrying, followed by dissolution and TTA extraction, using the "built-in" tracer ^{234}Th-Pa to measure recovery. The recovered thorium-230 (if any) can be determined by alpha counting, and the thorium-232 by neutron activation analysis.

C. Radioisotope Safety

1. Classification of $^{241}AmO_2$ Ring Source

A prototype $^{241}AmO_2$ ring source which was doubly encapsulated in stainless steel tubing has passed the test requirements for ORNL Class III-C. The inner and outer walls of the tubing were 0.030 and 0.020 in. thick, respectively. Both capsules were sealed with fusion welded stainless steel end caps. The source was fabricated in the shape of a ring having a 3-in. diameter. An earlier design of this source in which the activity was singly encapsulated in aluminum tubing was classified at a less demanding classification (II-B) due to the lower melting point of aluminum (1220°F) and the inability of the source to pass the Class III impact test.[4]

The following is a summary of the Class III-C tests performed on the stainless steel capsule. The vacuum leak test was negative after all the tests.

Operating Temperature Test - The source was immersed in dry ice (-70°F) for 24 hr then heated in air at 500°F for 24 hr.

Thermal Shock Test - The source was heated to 500°F, immersed in ice water (32°F) and then placed in dry ice (-70°F) for 15 min.

Maximum Temperature Test - The source was heated in air at 1700°F for 1 hr.

External Pressure Test - The source was hydrostatically tested in a pressure vessel at 1000 psig for 15 min.

Crushing Force Test - A 2000-lb force was applied to the source for 1 hr.

Puncture Test - The source was positioned on the 1/8-in.-diam puncture pin and a 70-g weight dropped on it from a height of 5 ft.

Shear Strength Test - The source was placed in the shear blocks and a 1000-lb force was applied for 1 hr.

Impact Test - The source was placed on an impact pad and a 12-lb weight was dropped on it from a height of 5 ft.

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[2]R. Shor, R. H. Lafferty, Jr., and P. S. Baker (eds.), *Strontium-90 Heat Sources*, ORNL-IIC-36, Oak Ridge National Laboratory (May 1971).
[3]J. A. Carter, *Anal. Lett.* (in press).
[4]A. F. Rupp, *Radioisotope Program (8000) Progress Report for May 1971*, ORNL-TM-3461, Oak Ridge National Laboratory, pp. 18-19.

TECHNOLOGY UTILIZATION - 08-01-05

A. Information Center

In August, 96 requests for information were filled with dispatch of 280 items, approximately 50% of which had already been prepared in the IIC and 40% depended on IIC personnel general background information. Approximately 350 IIC and TPR flyers and announcements of 600-word summaries were mailed to attendees of NBS Analytical Symposium. A list of reviews in progress is shown below.

Title	Author(s)	Status (% Complete)
Iodine-125	P. S. Baker and Martha Gerrard	75
Patent Literature on Process Radiation and Irradiator Design, Part 2. British and Canadian Patents, 1950 through 1970	R. E. Greene, Helen S. Warren, and P. S. Baker	38
Potato Sprout Inhibition by Radiation, Part 2	F. E. McKinney	50
Selected Abstracts of World Literature on Production and Industrial Uses of Radioisotopes, Part 3 of 1971	Martha Gerrard and P. S. Baker	10
Self-Diffusion in Liquids	F. J. Miller	75
Technetium-99m: Preparation and Uses	Martha Gerrard and P. S. Baker	65
Radioisotopes in the Textile Industry	F. J. Miller	95
Grain Disinfestation — A Worldwide Review	F. E. McKinney	20
Presowing Irradiation of Seed	Russian book translated and edited by Martha Gerrard	Final copy being proofed
Iodine-131 Production Methods	French report being translated by Martha Gerrard	Reference checking almost complete
Radioelectrochemistry	Helen P. Raaen	Final draft proofed
Nuclear Methods in Study of Environment	Helen P. Raaen	95
Fruit and Vegetable Bibliography (revision)	F. E. McKinney	90
Radioisotope Briefings for Industry — Minerals	R. H. Lafferty	80
Semiconductors	H. P. Raaen	In-progress
Special Sources (supplement)	F. E. McKinney	75
Bibliography of DID Contractor Publications	P. S. Baker	60
Use of Isotopes in Monitoring and Control of Environment Pollution	W. E. Mott and Martha Gerrard	75
Effect of Gamma Radiation on Nonalcoholic Drinks and Wine — Brandy Products (Russian book)	Martha Gerrard and P. S. Baker	Final typing being proofed
Isotope Education Experiments	Rhoberta Shor	50
List of AEC Radioisotope Customers, FY 1971	Ruth Curl (compiler)	60

B. Isotopes and Radiation Technology

Galley proofing for *Isotopes and Radiation Technology* 9(2) and writing and editing for 9(3) were continued.

C. Publications

P. S. Baker, A. F. Rupp, and Associates, *Isotopes and Radiation Technology* 8(4) (Summer 1971).

Martha Gerrard and P. S. Baker, *Selected Abstracts of World Literature on Production and Industrial Uses of Radioisotopes*, ORNL-IIC-37 (Part 1) (August 1971).

The following articles were published in *Isotopes and Radiation Technology* 8(4) (Summer 1971):

Martha Gerrard (summarized), Nuclear Techniques and the Fight Against Pollution

Martha Gerrard and F. E. McKinney (summarized), Activation-Analysis and Sulfur-Isotope-Ratio Techniques for Oil-Slick Identification

Martha Gerrard (summarized), Radioisotopic Sand-Tracer Study

Martha Gerrard, Radioisotope Gages in Pollution Control

Martha Gerrard and R. H. Lafferty (summarized), Kryptonate-Based Instrument for Detecting Automobile-Exhaust Pollutants

Martha Gerrard, Sewage and Waste-Water Processing with Isotopic Radiation: Survey of the Literature

Martha Gerrard (summarized), Conceptual Design of an Irradiation Test Facility for Waste Water and Sewage Sludge

R. H. Lafferty, P. S. Baker, and Martha Gerrard (summarized), Measurement of Sulfur Dioxide in Stack Gases: Design, Fabrication, and Test of Demonstration Instrument

Helen P. Raaen (summarized), Comparison of Radioisotopes and Fluorescent Dyes for Estuary Pollution-Control Studies

RADIOISOTOPE SALES

An order was received from Teledyne Isotopes for the encapsulation of 220 W of strontium-90 to be supplied by the U. S. Navy. A request for quotation was received from Teledyne Isotopes for a 350-W (∿51 kCi) strontium-90 source.

Shipments made during the month include 6,000 Ci of tritium to New England Nuclear Corporation, 10,000 Ci of tritium to Radium-Chemie A. Zeller and Company, 2,000 Ci of tritium to Canrad Precision Industries, 1 Ci of tellurium-123m to Bell Telephone Laboratories, 1,500 Ci of cesium-137 to Atomic Energy of Canada Limited, 500 Ci of cesium-137 to Radiochemical

Centre, England, 10,000 Ci of cesium-137 to U. S. Atomic Energy Commission, 2,000 Ci of cobalt-60 to University of California, Livermore, and 100 Ci of krypton-85 to Sumitomo Shoji Kaisha Limited, Japan. Other shipments were 66.36 Ci of xenon-133, 222 Ci of krypton-85, 20 Ci of 24.2% enriched krypton-85, 6 Ci of 26.5% enriched krypton-85, 4.1 μCi of plutonium-236, ∿0.2-mCi strontium-90 source, ∿4.5 x 10^{-3} μCi of iron-55, ∿35 mCi of cobalt-56, and 2,400 Ci of cobalt-60.

The radioisotope sales proceeds and shipments for July 1970 and 1971 are given in Table 10.

Table 10. Radioisotope Sales and Shipments

Item	July 1970	July 1971
Inventory items	$41,311	$36,059
Major products	9,110	4,715
Radioisotope services	12,096	8,905
Cyclotron irradiations	10,326	10,987
Miscellaneous processed material	2,623	6,715
Packing and shipping	6,480	6,012
Total	$81,946	$73,393
Number of shipments	241	218

ADMINISTRATIVE

Travel by IDC personnel and visitors to the IDC are given in Tables 11 and 12.

Table 11. Travel by IDC Personnel

Site Visited	Purpose of Visit
Tilton, New Hampshire	Attend Gordon Research Conference on Thin Films
Boston, Massachusetts	Attend 1971 Intersociety Energy Conversion Engineering Conference
Nashville, Tennessee	Participate in Spectroscopy Course at Fisk University
Richland, Washington	Attend Symposium on Packaging and Transportation of Radioactive Materials
Denver, Colorado	Attend ANS Conference on Applications of X-Ray Analysis

Table 11. Visitors to IDC

Visitor (affiliation)	Subject Discussed
U. S. Army Corps of Engineers	RIST program for FY 1972 and training program
Vanderbilt University Medical Center	Americium-241, xenon-133, and gadolinium-153 sources
Teledyne Isotopes	Strontium-90 heat sources
AEC, Washington	Division of Space Nuclear Systems programs
University of Tennessee	Uses of manipulators
U. S. Ambassadors to IAEA	Tour of Isotopes Development Center
Participants of IAEA course in use of tracer techniques in industrial and environmental pollution	Tour Isotopes Information Center
Representatives of five Negro colleges	AEC research and development contracts and use of Isotopes Information Center
Institute of Atomic Physics Bucharest, Romania	Cost benefit information on isotopes that would justify setting up a production facility in Romania
University of Tennessee Library	Tour Isotopes Information Center
St. Edwards University Austin, Texas	Tour Isotopes Information Center

PUBLICATIONS

JOURNALS

L. C. Brown, Determination of Phosphorus-32 and -33 in Aqueous Solution, *Anal. Chem.* 43, 1326 (1971).

C. V. Flanigan, M. A. Holsches, N. C. Dyer, L. C. Brown, and A. B. Brill, Experimental Model for Evaluation of Tumor Localizing Radiopharmaceuticals, *J. Nucl. Med.* 12(6), 355-6 (June 1971).

D. J. McMillan, J. H. Hamilton, and J. J. Pinajian, Energy Levels in ^{156}Gd Populated by 5.4-Day ^{156}Tb, *Phys. Rev. C* 4(2), 542-65 (August 1971).

REPORTS

J. H. Gillette, *Isotope Program (5000) Progress Report for Quarter Ending June 30, 1971*, ORNL-TM-3529, Oak Ridge National Laboratory.

Eugene Lamb, *Isotopic Power Fuels Monthly Status Report for July 1971*, ORNL-CF-71-8-28, Oak Ridge National Laboratory.

J. J. Pinajian, *Report of Foreign Travel to Japan, Republic of China, The Philippines, Thailand, and Vietnam, April 15 — November 7, 1966*, ORNL-CF-71-7-27, Oak Ridge National Laboratory (August 1971).

A. F. Rupp, *Radioisotope Program (8000) Progress Report for July 1971*, ORNL-TM-3526, Oak Ridge National Laboratory.

R. D. Seagren, *Safety Analysis of ORNL Bulk Radioisotope Shipping Cask*, ORNL-TM-3506, Oak Ridge National Laboratory (August 1971).

R. D. Seagren (comp.), *Waste Management at Oak Ridge National Laboratory*, ORNL-CF-71-7-9, Oak Ridge National Laboratory (July 12, 1971).

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