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RADIOISOTOPE PROGRAM (8000)
PROGRESS REPORT FOR AUGUST 1972

A. F. Rupp

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ISOTOPES DEVELOPMENT CENTER

RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR AUGUST 1972

A. F. Rupp

Work Sponsored by
AEC Division of Applied Technology

SEPTEMBER 1972

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RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR AUGUST 1972

A. F. Rupp

RADIOISOTOPE PRODUCTION AND MATERIALS DEVELOPMENT

REACTOR-PRODUCED ISOTOPES — 08-01-01

A. Biomedical Radioisotopes

1. 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 and to establish cooperative programs with medical institutions to evaluate it. Potassium-43 is potentially useful for metabolic and clinical studies of blood flow, rejection of transplanted organs, and kidney function. The half-life of potassium-43 is 22.3 hr; gamma-rays are 0.373 and 0.617 MeV.

Two 10-mCi batches of potassium-43 were processed and shipped this month. The summer months have resulted in less demand, but we feel an obligation to keep clinical studies going. Some of the unassigned potassium-43 is being used in a cooperative study with the ORNL Medical Instrumentation Group (Dr. P. R. Bell) to develop improved imaging techniques for potassium-43.

A problem noted in recent processing has been the tendency for potassium-43 to elute from the ion-exchange column with less resolution and more tailing. In one batch the potassium-43 product even contained a trace of sodium-24 which normally should be completely removed. The problem was finally correlated with introduction of a particular resin lot and a new batch has been ordered. The problem is suggestive of resin breakdown and loss of cross-linking. This instance points up the importance of the detailed processing records that are kept.

2. Platinum-195m

Recent medical research has shown that some platinum compounds act as chemical therapeutic agents towards certain kinds of tumors. Platinum radioisotopes are needed for investigation of the therapeutic mechanism involved. Platinum-195m (4.0 d) has gamma emissions of 99 keV (11%) and 129 keV (2.8%) that make it suitable for whole-body scanning.

Two shipments of platinum-195m were made to Dr. Walter Wolf, USC, under our cooperative program. He reports that they have improved their yield in the synthesis of the cis-dichlorodiammine-^{195m}Pt(II). He also reports that they have begun a long-term study of tumor-infected mice after treatment with the radiopharmaceutical, and they have established that after the initial stage of rapid clearance of the drug there remains a considerable long-term localization of the platinum-195m in the tumor.

Two samples of recently enriched batches of platinum-194 were irradiated in the HFIR. The single-pass enrichments were >96%. The data from the irradiation are presently being analyzed to determine impurities and to determine what improvement in platinum-195m specific activity can be expected.

3. Process Assistance — (4.53-d) Calcium-47

Calcium-47 is now being produced at higher specific activity in the HFIR than formerly when only the ORR was used, and the procedure of precipitating $^{47}CaCO_3$ with Na_2CO_3 to remove sodium-24 activity is no longer satisfactory. A cation-exchange procedure was developed to load the calcium-47 onto the resin bed from neutral solution, elute sodium-24 with 0.2 *M* HCl, and then strip the calcium-47 product with 5 *M* HCl. In testing the procedure, we noted that several trace radiocontaminants in addition to sodium-24 were also removed in the process: principally cadmium-115 (and indium-115m daughter), tungsten-187, and potassium-42. The yield of calcium-47 is approximately 95%.

4. Production of Medical-Grade Plutonium-238 in a Reactor Loop

Use was made of existing computer programs to calculate an ideal production rate of plutonium-238 from americium-241 using a closed loop irradiation and recovery scheme. The data were compared directly with previous reports on a batch irradiation-batch processing method. The comparison shows that the percentage increase in plutonium-238 production with a loop process increases with increasing neutron flux. The calculated percentage increases for a reactor loop over a batch irradiation are:

Flux (n/cm^2·sec)	Increase (%)
1.84×10^{14}	11.42
3.0×10^{14}	15.36
5.5×10^{14}	31.2

5. Phosphorus-33

Two methods for producing phosphorus-33 low in phosphorus-32 are being evaluated: irradiation of enriched sulfur-33 (>92 at. %) or chlorine-36 (∿63 at. %) with fast neutrons. 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}).

A 0.85-g >90% enriched $K_2{}^{33}SO_4$ target was processed after irradiation for five cycles in the HFIR. Upon opening the target assembly the quartz ampul was found to be broken, and the target had to be washed out of the target capsule. The yield of phosphorus-33 was approximately 250 mCi, half what would normally be expected for this target. The phosphorus-32 content was 6%, indicating no serious isotopic degradation of the target. The recovery of enriched sulfur-33 is in progress, and process equipment and residues are being checked to determine whether phosphorus-33 was held up at some intermediate step.

A question of a consistent bias between process analyses and product analyses was investigated at length. The bias is generally less than 10%, but a customer complaint about a product assay led to a study of the problem. New standards of phosphorus-32 and calcium-45 (the stand-in for phosphorus-33) were ordered and a comparison of techniques and check of system efficiencies and stabilities are in progress.

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

Processed Units		Service Irradiations	
Radioisotope	Amount (mCi)	Type	Number
Copper-67	22	Platinum-196	2
Calcium-47	22	MoO_3	1

ACCELERATOR-PRODUCED ISOTOPES — 08-01-02

A. Cyclotron Products Pilot Production
(Production and Inventory Accounts)

August 1972 ORNL 86-Inch Cyclotron runs for ORNL and non-ORNL programs are given in Table 1.

Table 1. ORNL 86-Inch Cyclotron Runs for August 1972

Product	No. of Runs	Time (hr:min)	Total Charges
	ORNL Programs		
Cobalt-61	4	11:30	$1,120
Gallium-67	2	18:25	1,764
		29:55	$2,884
	Non-ORNL Programs		
Cobalt-57	1	51:15	$8,581

FISSION PRODUCTS — 08-01-03

A. Krypton Enrichment

The three thermal diffusion units loaded with krypton are continuing to build up the mass 85 activity in the center of the system and show no signs yet of reaching equilibrium.

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

1. Processing and Process Status

One cesium oxalate process cycle (three batches) was run, and the resulting cesium carbonate was converted to $^{137}CsCl$ in eight batches. The estimated yield was 75,000 Ci of product. Alum accumulation of about 200,000 Ci of cesium-137 for another oxalate-chloride cycle is in progress. Ten special-form cans of calcined cesium oxalate were loaded for encapsulation.

A considerable inventory of cesium-137 glass residue, resulting from processing done several years ago, is on hand. All of this material was decanned, crushed, and combined into one fraction (about 6 kg containing an estimated 75,000 Ci). An attempt will be made to recover some of the cesium-137 by a simple leaching/filtration process. This will be done in small batches using laboratory-sized equipment and will be a low-priority item. Any recovered cesium-137 will be recycled to the cesium-137 feed stream for purification by alum crystallization.

The current cesium-137 process status is shown below.

Item	Cesium-137 (Ci)
In-process material	733,000
$^{137}CsCl$ products	192,500
Sources in fabrication	0
Completed sources awaiting shipment	182,500

2. Operational Summary

Item	August 1972		CY 1972		FY 1973	
	No.	Ci	No.	Ci	No.	Ci
HAPO shipments received	0	0	0	0	0	0
Product batches prepared	8	75,000[a]	16	148,300[a]	15	141,300[a]
Sources						
Fabricated	0	0	23	55,300	0	0
Shipped	0	0	23	55,300	0	0
Special form cans						
Fabricated	10	500	20	114,400	10	500
Shipped	1	10	7	1,400	2	500

[a]Estimated; analysis of August production incomplete.

3. Current Orders

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

Customer	Amount (Ci)	Estimated Shipping Date
Brookhaven National Laboratory	203,000	FY 1973
Lockheed Georgia Company	∿35,000	a
Radiochemical Centre, England	400	September 1972
Minnesota Mining & Mfg. Co.	500	September 1972
Technical Operations, Inc.	8	September 1972
J. L. Shepherd Associates, Inc.	50,001	To be scheduled

[a]Sources are in storage awaiting receipt of customer's container.

An order is on hand from Atomic Energy of Canada Limited for ∿113,000 Ci of cesium-137 as CsCl powder. The powder has been canned and stored awaiting release by customer.

4. Source Fabrication

The following powder shipments were made during August 1972:

Customer	Isotope	No. Cans	Total Curies
General Nuclear	Cesium-137	1	10
Radiochemical Centre[a]	Cesium-137 Carbonate	10	500

[a]The shipment of cesium carbonate is being delayed pending the arrival of a fire shield for the carrier.

During August, using a batch of 66,000 Ci of ^{137}CsCl, work was begun on an order of sources containing a total of about 180,000 Ci for the Brookhaven National Laboratory.

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

1. Processing and Process Status

No strontium-90 processing was done this month. The current strontium-90 process status is as follows.

Item	Strontium-90 (Ci)
In-process material	805,900
Strontium-90 products	426,200
Sources in fabrication	0
Returned SNAP sources	446,600
Completed sources awaiting shipment	221,000

2. Operational Summary

Item	August 1972		CY 1972		FY 1973	
	No.	Ci	No.	Ci	No.	Ci
HAPO shipments received	0	0	0	0	0	0
Product batches prepared	0	0	13	510,000	0	0
Sources						
Fabricated	0	0	9	581,000	0	0
Shipped	0	0	5	360,000	0	0
Special form cans						
Fabricated	0	0	39	5,600	0	0
Shipped	0	0	13	900	0	0

3. Current Orders

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

Customer	Amount (Ci)	Estimated Shipping Date
U.S. Navy (ORNL)	221,000	FY 1973
U.S. Navy	278,675	a

[a]To be released by customer.

4. URIPS-8 Generators

Load test readings of the four URIPS-8 generators are continuing. Typical readings for the four generators are given in Table 2.

Table 2. URIPS-8 Generator Test Data

Generator Number	Load		Shorted		Temperature (°F)	
	Volts	Amps	Volts	Amps	Fin	Hot Shoe
25	5.22	1.91	0.04	3.40	123	518
26	5.10	1.89	0.03	3.30	125	523
27	5.09	1.86	0.04	3.36	130	512
28	5.09	1.86	0.02	3.45	122	482

The Navy has requested that we continue daily monitoring of generator performance through September 22. Discussions have been held with representatives of the Division of Materials Regulation with regard to obtaining a shipping license for the generators. It was argued that the ORNL-developed Heating III computer code could be used for redoing the thermal analysis. Work is in progress to program the needed data into the ORNL computer.

D. Promethium Melting Point and Phase Transition

The melting point of promethium metal prepared by the vacuum-reduction-evaporation technique was measured to be 1037°C. The same melting point was observed for each of three promethium samples in separate determinations. Calibrations were performed using silver and gold under the same circumstances as the actual promethium melts. Each of the samples rested on a MgO container, and the occurrence of melting was observed visually.

The melting point of one piece of promethium metal that was left in air overnight was observed to be 1063 ± 5°C. It is believed that the presence of Pm_2O_3 increased the melting point of the sample.

Another promethium metal sample was placed on a W5Re-W26Re thermocouple, which was connected to a similar thermocouple to form a difference couple. A Pt-Pt10Rh thermocouple in the vicinity of the difference couple was used to monitor the temperature. By running both heating and cooling curves on this sample, the melting point and freezing point was observed to be 1046°C.

A phase transition in promethium metal was observed in a similar manner. The transition occurred at 890°C during heating or cooling. This phase transition should be the hexagonal-to-bcc phase transition as also found in Pr, Nd, Gd, etc.

Another experiment was performed with a promethium sample on a Pt-Pt10Rh thermocouple. On heating the sample at a constant rate, the output of the couple showed definite evidence of a thermal arrest at 900°C; also the heating rate increased above this temperature. From this we infer that the heat capacity of the bcc form is less than the hexagonal form in the vicinity of the phase transition.

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

Isotope	Number of Batches	Amount (Ci)
Xenon-133	2	700
Iodine-131	1	28
Zirconium/Niobium-95	1	a
Barium-140[b]	1	6
Strontium-89[b]	1	24

[a]Analysis not available.

[b]Runs made and reported during July; analysis has now been reported.

F. Promethium-147 Shipments and Current Orders

Donald W. Douglas Laboratories' order for 51,000 Ci of promethium-147 will be shipped in October 1972. A 5000-Ci shipment of promethium-147 was made to 3M Company.

G. Removal of Krypton-85 from Fission-Produced Xenon

Several batches of fission xenon have been processed for krypton-85 removal by using the existing krypton-85 gas chromatograph purification system. A total of 2450 liters of gas (STP) has been collected. The quantity and purity of the collected gas are shown below.

Cylinder Number	Krypton-85 (μCi/liter)	Xenon (liters)
1	0.18	236
2	0.159	366
3	0.047	402
5	0.138	489
8	0.914	97
9	0.492	51
12	0.199	366
13	0.072	443

Mass analysis on cylinder 1 indicated a chemical purity of 99.93% with ∿0.03% mass 28. The mass 28 (N_2 or CO) is believed to be N_2 since the gas was chemically purified by passing it through a heated copper oxide trap followed by exposure to anhydrous magnesium diborate. A second cylinder was purified with the same system and yielded a 99.72% chemically pure product. Removal of residual nitrogen can be accomplished by treatment of the gas with molten calcium.

SOURCE DEVELOPMENT — 08-01-01

A. Aluminum Foil Krypton-85 Sources

The reconstructed vacuum chamber for this project is now an all-glass system except for the copper electrodes used to hold the foil disks to be bombarded. Leaks have been eliminated, and outgassing problems have been controlled to give much more reliable results.

Aluminum foil disks have been bombarded with normal fission krypton containing 4.5% mass 85, and the surface activity achieved varies between 200 and 250 mR/hr. Disks made with depleted 0.7% krypton-85 read about 9 mR/hr.cm^2, and those made with 4.5% krypton-85 read 64 mR/hr.cm^2. This indicates that the surface activity achieved is directly proportional to the concentration of krypton-85 in the gas used, and foils of still higher activity can be made by using enriched krypton.

B. Radioisotope Characterization, Quality Control, and Standards

1. Radioisotope Characterization

Barium-133 is useful as a long-lived standard for measurements of intensities of photons of low to moderate energies (0.4 MeV). Several careful studies have been made of its decay scheme, and one[1] of them disagreed with the others. An error was found in the Notea and Gurfinkel calculations; correction resulted in agreement with other values, including those listed in the *Isotope User's Guide*. The x-ray intensity is still uncertain, however, and further study is in progress.

Inquiries were handled concerning the cross section of ytterbium-168, the names of some medical researchers interested in nuclear data, and general references on radiochemistry and radioisotopes, for which we furnished an updated 1971 list.

2. Radioisotope Special Analysis and Quality Control

S. A. Reynolds has been appointed chairman of ANS-16, the Standards Committee of the ANS Isotopes and Radiation Division. Members are being sought, and plans are being developed for the committee's work. Early tasks are expected to be concerned with standard materials and methods for radioisotopes, including large sources, and standard decay-scheme data. A meeting is scheduled for November 14 in Washington.

Routine tritium analytical procedures were reviewed and were found to be appropriate, except that some organic samples may have had low results because of the fact that water was present, unknown to the analysts. Such samples can be assayed by addition of a tritium-toluene standard, since more toluene does not affect efficiency of the system.

There were inquiries on the characteristics of reagent H_2O_2, analytical methods for organic acids, absorption of beta radiation in mixed absorbers, names of specialists in source-measurement techniques, and uranium content of soils (typically a few parts per million).

RADIOISOTOPE SALES

ITEMS OF INTEREST

An order for 50,001 Ci of cesium-137 as bulk powder for storage and sources was received from J. L. Shepherd Associates, Inc. An inquiry was received from Douglas Aircraft Corporation for a 1500-Ci strontium-90 source.

Shipments made during the month that may be of interest are listed below.

---

[1] A. Notea and Y. Gurfinkel, *Nucl. Phys.* A107, 193-208 (1968).

Customer	Isotope	Amount
Large Quantities		
CEA, France	Iodine-129	1 g
Minnesota Mining & Manufacturing Company	Promethium-147	5,008 Ci
New England Nuclear Corporation	Tritium	4,000 Ci
U.S. Radium Corporation	Tritium	10,000 Ci
Radium-Chemie A. Zeller & Company	Tritium	10,000 Ci
Radium-Chemie A. Zeller & Company	Promethium-147	300 Ci
Items Shipped for Use in Cooperative Programs		
Oak Ridge Associated Universities	Gallium-67	
University of Southern California	Platinum-195m	
Withdrawn Items		
Mayo Clinic	Copper-67	2 lots
Cleveland Metropolitan General Hospital	Iodine-131	50 mCi
University of Rochester	Iodine-131	100 mCi
University of Pittsburgh	Iodine-131	50 mCi
University of California, Berkeley	Iodine-131	30 mCi

A summary of radioisotopes shipped during July 1972 is given in Table 3.

Table 3. Summary of Radioisotopes Shipped During July 1972

Radioisotope	Number of Shipments	Quantity (mCi)
Cesium-137	1	500,000
Strontium-89	2	7
Cerium-144	1	5
Promethium-147	2	655,000
Krypton-85, enriched	4	112,000
Krypton-85, normal	10	303,110
Iodine-129	6	2,725[a]
Gross fission products	1	1
Zirconium-95	1	5
Niobium-95	4	20
Praseodymium-143	1	4
Ruthenium-103	4	6
Ruthenium-106	4	7
Yttrium-91	1	10
Xenon-133	55	75,670
Iodine-131	7	410
Argon-37	3	21
Europium-152-154	1	2
Phosphorus-33	3	102
Strontium-90	10	500

Table 3. continued

Radioisotope	Number of Shipments	Quantity (mCi)
Tritium	10	5,192,000
Experimental products	8	30
Copper-67	4	26
Gallium-67	5	212
Gadolinium-153	1	50
Indium-111	1	50
Potassium-43	4	8.5
Service Irradiations		
Cyclotron	2	
Reactors	2	
	158	6,839,256.5
		2,725[a]

[a]Milligrams

The radioisotope sales proceeds and shipments for the first month of FY 1972 and of FY 1973 are given in Table 4.

Table 4. Radioisotope Sales and Shipments

Item	7-1-71 thru 7-31-71	7-1-72 thru 7-31-72
Inventory items	$36,059	$18,650
Major products	4,715	4,340
Cyclotron irradiations	10,987	9,194
Radioisotope services	8,905	10,920
Miscellaneous processed material	6,715	6,604
Packing and shipping	6,012	5,070
Total	$73,393	$54,778
Number of Shipments	218	158

ADMINISTRATIVE

Visitors to the IDC are given in Table 5.

Table 5. IDC Visitors

Affiliation	Subject Discussed
Boris Kidrich Institute, Yugoslavia	Production of radioisotopes
University of Tokyo, Japan	Radioisotope processing
Teledyne Isotopes	Plans on curium-244

PUBLICATIONS

REPORTS

D. N. Braski and B. F. Early, *Preparation of Self-Supported GeO_2–16% P_2O_5–8% U_2O_5 Films by Sputtering*, ORNL-TM-3830, Oak Ridge National Laboratory (August 1972).

Eugene Lamb, *Isotopic Power Fuels Monthly Status Report for June 1972*, ORNL-TM-3920, Oak Ridge National Laboratory.

J. H. Gillette, *Isotopes Program (5000) Progress Report for Quarter Ending June 30, 1972*, ORNL-TM-3919, Oak Ridge National Laboratory.

A. F. Rupp, *Radioisotope Program (8000) Progress Report for July 1972*, ORNL-TM-3942, Oak Ridge National Laboratory.

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