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RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR DECEMBER 1972

A. F. Rupp

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ISOTOPES DEVELOPMENT CENTER

RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR DECEMBER 1972

A. F. Rupp

Work Sponsored by
AEC Division of Applied Technology

JANUARY 1973

[redacted]

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RADIOISOTOPE PROGRAM (8000) PROGRESS REPORT FOR DECEMBER 1972

A. F. Rupp

RADIOISOTOPE PRODUCTION AND MATERIALS DEVELOPMENT

REACTOR-PRODUCED ISOTOPES — 08-01-01

Silicon-32 *(T. A. Butler)*

Silicon-32 (~650 year) exhibits many desirable physical decay characteristics that make it an interesting and attractive radionuclide for a variety of uses: (1) the only long-lived radionuclide of silicon for use in agriculture, geology, solid-state physics, etc., (2) as a parent for a ^{32}Si-^{32}P generator system which would offer a constant supply of phosphorus-32 for medical and scientific application, and (3) as a long-lived Cerenkov counting standard for phosphorus-32 determinations. Reactor production of silicon-32, which will be investigated initially, can occur via three completely different reaction paths: (1) $^{30}Si(n,\gamma)^{31}Si(n,\gamma)^{32}Si$, (2) $^{31}P(n,\gamma)^{32}P(n,p)^{32}Si$, and (3) $^{34}S(n,\gamma)^{35}S(n,\alpha)^{32}Si$. Silicon-32 can also be prepared in large quantities via the spallation reaction using high-energy (>100 MeV) protons and will be investigated in collaborative efforts with other AEC laboratories as high currents of these high energy protons become available.

The long-cooled $AlPO_4$ sample reported on earlier,[1] that had been irradiated in the HFIR for four cycles (the earlier reference to three-cycle irradiation of this target was incorrect), was opened and the target material was quantitatively dissolved in 10% NaOH (which should offer a stable medium for carrier-free $^{32}SiO_3^{2-}$ ion). A high-resolution gamma-ray spectrum of this solution, obtained by using a Ge(Li) detector connected to a 4096-channel pulse height analyzer, showed cobalt-60, antimony-124, and antimony-125 as the major gamma-emitting radiocontaminants present. Due to the presence of appreciable amounts of these radiocontaminants, direct Cerenkov radiation counting[2] of an aliquot of the basic dissolver solution could not be used to determine the phosphorus-32 in secular equilibrium with the silicon-32. In order to determine the silicon-32 produced in the target, known aliquots of the basic dissolver solution were processed via the strong cation-exchange, weakly basic anion-exchange chromatographic procedure developed earlier.[1] A double tracer technique using short-lived $^{31}SiO_3^{2-}$ (2.62 hr) was used

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[1]A. F. Rupp, *Radioisotope Program (8000) Progress Report for September 1972*, ORNL-TM-4002, p. 2.
[2]L. C. Brown, *Anal. Chem.* 43, 1326 (1971).

to determine chemical yields of the separation process, and the silicon-32 was calculated from the in-growth of phosphorus-32 into the freshly separated silicate fraction. The silicon-31 and phosphorus-32 activities were followed by Cerenkov radiation counting of slightly basic aqueous samples, but small amounts of cobalt-60 were detected which resulted in a low-level, long-lived Cerenkov radiation background activity which was <10% of the phosphorus-32 secular equilibrium activity.

The data indicate a total silicon-32 content in this four-cycle LFIR irradiated $AlPO_4$ target of approximately 0.97 μCi, which is probably the largest amount of carrier-free silicon-32 produced by man to date. Based on this preliminary yield, the silicon-32 content of the other $AlPO_4$ target that had been prepared at the same time as this four-cycle target, but which has been irradiated for a total of 18 cycles (41,016 MW'd over approximately 15 months), should be approximately 5 μCi. This larger amount will be used for the investigation of the physical decay properties of this poorly characterized radionuclide.

The data from this four-cycle target have also yielded a preliminary estimate for the ratio of the half-life of silicon-32 (in years) to the (n,p) cross section for the $^{32}P(n,p)^{32}Si$ reaction (in barns) of approximately 530. If one then uses the empirical approach of Roy and Hawton[3] to estimate the $^{32}P(n,p)^{32}Si$ cross section as approximately 465 mb, then a half-life value of approximately 250 years is obtained for silicon-32, in good agreement with the work of Honda and Lal.[4] Future work will emphasize (1) continued chemical process development to eliminate all cobalt-60 and antimony-124,125 contamination of the final product and (2) direct measurement of the half-life of silicon-32 by mass spectrometry and counting, i.e., by specific activity.

Phosphorus-33 *(T. A. Butler)*

The purpose of this project is to develop methods of preparing hundred-millicurie quantities of carrier-free phosphorus-33 containing ≤ 5% phosphorus-32. The two methods for 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 organophosphorus compounds, and double labeling experiments.

---

[3] J. C. Roy and J. J. Hawton, *Table of Estimated Cross Sections for (n,p), (n,α), and (n,2n) Reactions in a Fission Neutron Spectrum*, CRC-1003 (December 1960).

[4] M. Honda and D. Lal, *Nucl. Phys.* 51, 363 (1964).

An 0.80-g $K_2{}^{33}SO_4$ target (>90%) was processed, and the phosphorus-33 was combined with material from previous targets which had had high phosphorus-32 content and had been allowed to decay. The $K_2{}^{33}SO_4$ target was recovered with a yield of 92%, reencapsulated, and reinserted in the reactor.

A 0.42-g $K^{36}Cl$ target, from which the chlorine-36 had been recovered by the AgCl procedure, was processed. The phosphorus-32 content was <5% at time of processing, although the yield was only half what was expected by the irradiation of a 0.42-g target. The target was dissolved in water and passed through a hydrogen form cation-exchange column to convert to $H^{36}Cl$. Iron carrier was added by dissolving freshly precipitated $Fe(OH)_3$ in the HCl solution and then reprecipitating the $Fe(OH)_3$ with cesium-free KOH.

Potassium-43 *(T. A. Butler)*

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 22.5 hr; gamma-rays are 0.373 and 0.617 MeV.

Two batches of potassium-43 were processed in November and three more were processed in December. Over 200 mCi has been delivered this fiscal year.

Platinum-195m *(T. A. Butler)*

The objectives of this work are to develop methods for preparing platinum isotopes suitable for whole-body scanning in biological and medical experiments, to characterize the products, and to establish cooperative programs with medical research groups interested in evaluating their usefulness.

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, one to Michigan State University and one to University of Southern California.

Test samples of >94% enriched platinum-194 were irradiated for 10 days in the HFIR. Whereas the present 57% enriched platinum-194 has ten times more platinum-197 activity than platinum-195m at discharge, the new preparation has less platinum-197 than platinum-195m at discharge, thus obviating the need for a decay period. The test samples taken prior to chemical purification showed the presence of cerium, lanthanum, and scandium impurities in the platinum target by their induced activities. Two grams of the stable target has been chemically purified and will be made available for medical applications in time for trials with human subjects that are scheduled at the University of Southern California.

ACCELERATOR-PRODUCED ISOTOPES — 08-01-02

Cyclotron Products Pilot Production (Production and Inventory Accounts) *(L. O. Love)*

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

Table 1. Cyclotron Irradiations and Runs for December 1972

Product	No. of Runs	Time (hr:min)	Total Charges
	ORNL Programs		
Cobalt-56	1	6:00	$ 703
Gallium-67	3	15:45	1,517
Lead-203	1	2:45	293
		24:30	$ 2,513
	Non-ORNL Programs		
Cobalt-57	2	102:30	$15,764

FISSION PRODUCTS — 08-01-03

Cesium-137 Pilot Production (Production and Inventory Accounts) *(R. W. Schaich)*

1. Process Status

A cesium carbonate preparation cycle which was started in November was completed, making a total of approximately 130,000 Ci of intermediate material available for conversion to ^{137}CsCl product. The ^{137}CsCl processing cycle was started; however, a suspected water leak in an underground

cell ventilation duct during heavy rains resulted in a shutdown of all operations. Three batches of $^{137}CsCl$ in various stages of completion were secured, and all the remaining carbonate was stored.

The remaining cesium-137 feed solution was brought into the crystallizer system, and a preliminary water rinse of all cesium-137 storage tanks was made in preparation for the final FPDL cleanup. The current cesium-137 process status is shown below:

Item	Cesium-137 (Ci)
In-process material	420,000
$^{137}CsCl$ products	301,100
Sources in fabrication	22,800
Completed sources awaiting shipment	182,500

2. Operational Summary

Item	Dec. 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	31	314,000	30	307,000
Sources						
Fabricated	20	25,400	49	81,000	26	25,700
Shipped	20	25,400	49	81,000	26	25,700
Special form cans						
Fabricated	0	0	20	114,400	10	500
Shipped	1	500	9	2,400	4	1,500

3. Current Orders

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

Customer	Amount (Ci)	Estimated Shipping Date
Brookhaven National Laboratory	203,000	FY 1973
J. L. Shepherd Associates, Inc.	24,754	January 1973

An order is on hand from Atomic Energy of Canada, Limited, for 88 kCi of cesium-137 as CsCl powder. The powder has been canned and stored, awaiting release by the customer.

4. Source Fabrication

Five hundred curies of cesium-137 powder was shipped to 3M Company. The cesium-137 sources shown on page 6 were shipped in December.

Customer	Type	Number of Sources	Total Curies
Technical Operations	Customer	6	150
AECL, Canada	Teletherapy	14	25,285

Strontium-90 Pilot Production (Production and Inventory Accounts) *(R. W. Schaich)*

1. Process Status

No strontium-90 processing was done. The current strontium-90 process status is shown below.

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	Dec. 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	14	900	1	10

3. Current Orders

The U. S. Navy has placed an order for 221,000 Ci of strontium-90 to be shipped in FY 1973.

4. URIPS-8 Thermal Analysis *(R. A. Robinson)*

A thermal analysis of the URIPS-8 generator using the HEATING-3 computer program[5] has been completed. The steady-state temperature profile was calculated using the ambient conditions specified in Title 49, *Code of*

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[5]W. D. Turner and M. Siman-Tov, *HEATING-3 — An IBM-360 Heat Conduction Program*, ORNL-TM-3208, 1971.

Federal Regulations, paragraph 173.393(e). Figure 1 is a section view of the generator with the steady-state temperatures indicated for various points. The maximum fuel temperature is 1282°F and the hot shoe temperature is approximately 529°F. The temperature gradient along the top surface is due to the cooling fins attached to the generator.

To analyze a system with the HEATING-3 code, the system must be divided into homogeneous sections having parallel faces. Each junction of section lines defines a node of the system. Heat may flow from a node to each adjacent node along paths which are parallel to each axis chosen to represent the system. The URIPS-8 generator is approximated by the system of nodes shown in Fig. 2.

The URIPS-8 generator was also evaluated for its ability to withstand a 30-min exposure to fire as required by Title 49, *Code of Federal Regulations*, paragraph 173.398(c)(2)(iii). The temperature profile calculated in the steady-state analysis was used for the initial conditions of the fire situation. Had the fin geometry been incorporated into the computer model geometry, the resulting computer computation time (CPU) for the fire condition would have been impractical (270 min). Therefore, fin equivalent heat transfer coefficients were calculated for boundary 17 of region 44 (see Fig. 2) to simulate the heat transfer characteristics of the fins. Preliminary calculations with the code indicated that the fins reached their melting point in approximately 15 min after the start of the fire. To simulate melting of the fins, the calculations were stopped after 15 min of the fire and the fin equivalent heat transfer coefficients were replaced with coefficients that reflected the boundary conditions which would exist without the fins. Latent heat of melting of the fins was negligible and was ignored; it was assumed that the fins maintained their initial geometry until 15 min into the fire at which time sudden melting occurred.

The thermoelectric module (region 34) was assumed to have a constant thermal conductivity for the entire time of the fire. The calculated temperature of the module after the first 30 min is 982°F. This is 81°F below the melting point of bismuth telluride. Melting of the thermoelectric module makes a significant difference as to how fast the fuel capsule is cooled and how high the fuel capsule temperature will go after the 30 min of the fire. To insure that the most severe case was considered, it was assumed that the thermoelectric module remained intact during the 30 min of the fire (thus conducting maximum heat into the system) and at the end of the 30 min, melting occurred and an argon-filled gap was formed (thus creating a greater resistance to heat flow out of the system during cool-down).

Figure 3 is a graphical presentation of the temperatures of several key nodes throughout the system for the 30 min of the fire and a 4-hr period afterward. Although the fuel capsule temperatures are still increasing, the increase is small (i.e., during the final 30 min the capusle temperature increase was 6°F). If it is assumed that the capsule experiences a maximum temperature of 1887°F, this is still 431°F below the Hastelloy C-276 melting range.

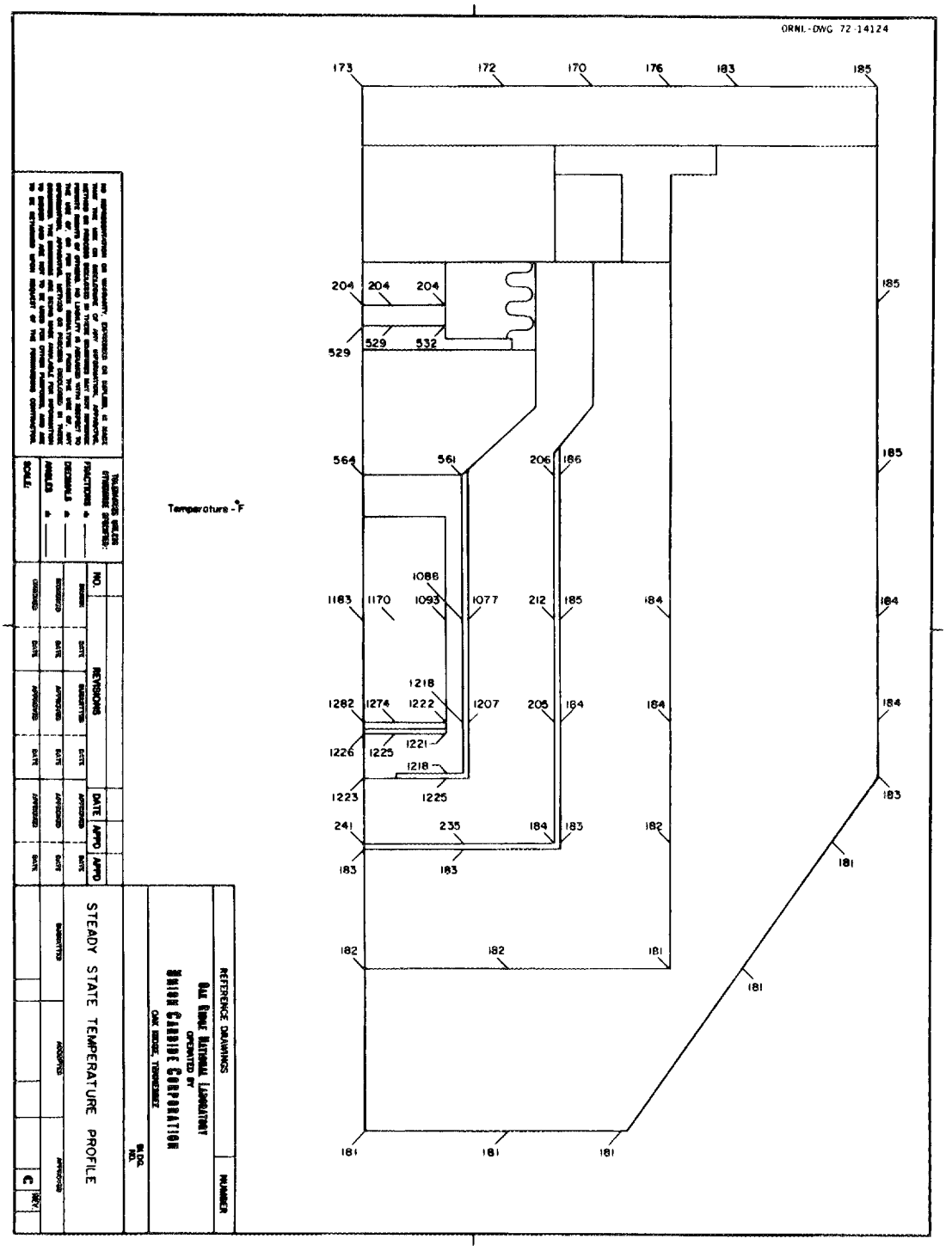

Fig. 1. URIPS-8 Steady-State Temperature Profile.

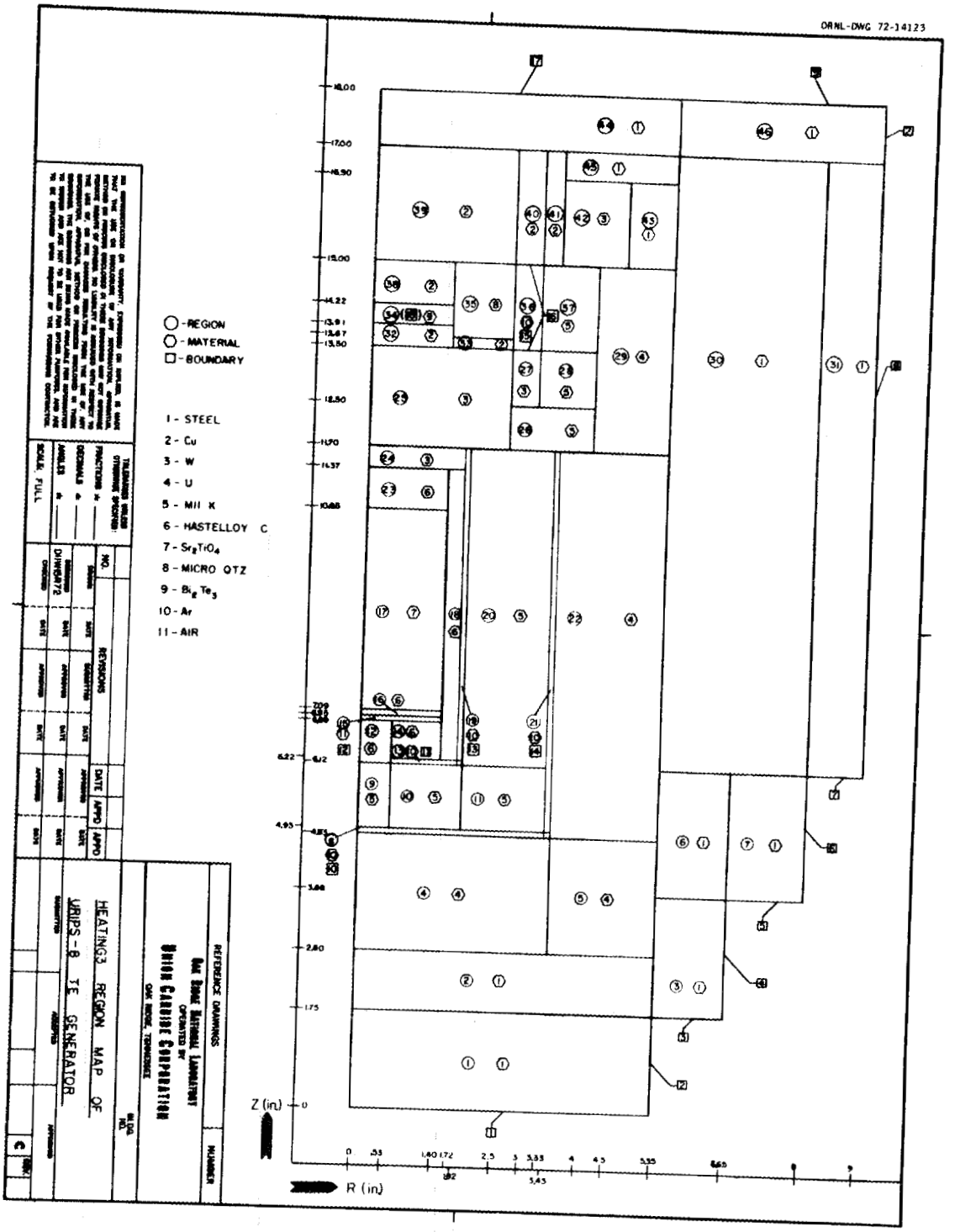

Fig. 2. HEATING-3 Region Map of URIPS-8 Generator.

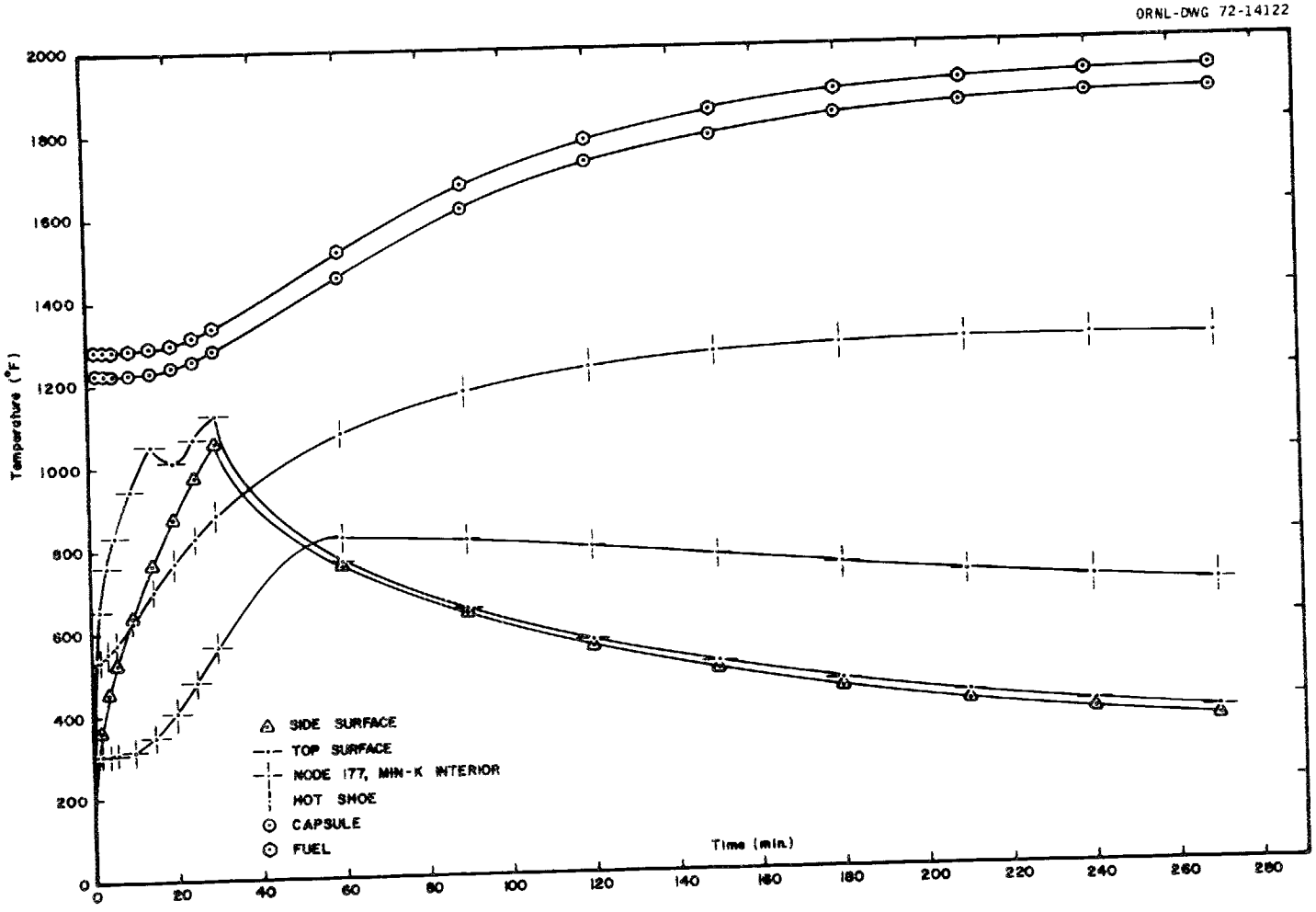

Fig. 3. URIPS-8 Fire Test Temperature Distribution.

Short-Lived Fission Products Pilot Production
(Production and Inventory Accounts) *(R. W. Schaich)*

Isotope	Number of Batches	Amount (Ci)
Iodine-131	1	∿59
Niobium-95	1	9
Ruthenium-103	1	41
Xenon-133	3	1000

SOURCE DEVELOPMENT — 08-01-04

Americium-241 *(F. N. Case)*

A cooperative effort between IDC and the Tuskegee Institute of Technology involves determining the photon output of americium-241 sources which are commercially available. In particular, the ratio of the quantity of the 27-keV and the 59-keV photons which emerge from the sources would give a method for the comparison of sources. Two americium-241 photon sources were examined, one from Monsanto Research Corporation and the other from The Radiochemical Centre, Amersham, England. Each source contained approximately 300 mCi of americium-241 and was fabricated with the same geometry, i.e., 1/2-in. diam and 1/2-in. length.

Measurements were made at various distances with a Li-Si detector and pulse height analyzer for the two sources. In addition to the studies which were made on the two commercial sources, measurements were made with a standard americium-241 source so that the photon energies lower than 27 keV could be examined. The data consisting of printout from the multichannel analyzer will be sent to Tuskegee for analysis and interpretation.

Radioisotope Characterization, Quality
Control, and Standards *(S. A. Reynolds)*

1. Radioisotope Characterization

This program is basically a supportive one, supplying data needed for practical purposes to those who analyze or use isotopes locally and in other institutions. Valid and readily usable nuclear data continued to be a major item of interest. Working through subcommittee ANS-16, we have contacted compilers of radiation energies and intensities, in addition to those in the U. S. Nuclear Data Committee and the Office of Standard Reference Data. Most of the needed data exist, but problems persist with respect to preparation in a convenient format and mechanism for revision.

An inquiry about the half-life of cesium-137 led to learning of mass-spectrometric values of 30.17 and 2.06 years for cesium-137 and cesium-134, respectively, which are in excellent agreement with our published values. Inquiries were answered on the specific gamma activity of natural uranium and the half-life of curium-248.

2. Radioisotope Special Analysis and Quality Control

The College of American Pathologists (CAP) is continuing its very useful program of intercomparisons of the assay of important radiopharmaceuticals in various laboratories. Local measurement of an iodine-125 standard prepared for CAP by NBS showed agreement within 1.1%, well inside the NBS certification of 1.8%. Another project, in which we did not participate, involved medical laboratories sending medical quantites of chromium-51 to NBS. The NBS values ranged from 60% to 368% of the values reported by the laboratories. An ORNL phosphorus-32 preparation was calibrated by NBS, with the finding that our routine laboratory value was within 0.1% of the NBS calibration. Measurement of xenon-133 is difficult because of the low energy of its gamma radiation and the high levels of activity which are dispensed. We have compared assays of samples with one of the major commercial suppliers and observed a persistent discrepancy of about 13%. An NBS measurement of a very dilute sample confirmed our assay, according to a verbal communication. We are continuing tests to confirm our measurement, since NBS personnel indicate difficulty in measuring xenon-133 at practical levels. We suggested microcalorimetry, which NBS has used for other standardizations. These are examples of our use of NBS standards and calibration services, and we continue to advocate nation-wide use of NBS expertise.

The recent meeting of ANS-16 was attended by six of fourteen members and seven representatives of other groups. Among items discussed were subcommittee scope, liaison with other standards organizations, technical sessions for future national ANS meetings, long-term gamma dosimetry, large-source measurements, xenon-133 assay, and nuclear data needs. Some liaison representatives have been appointed, and certain tasks have been assigned. We were asked by ANSI to nominate a representative to attend a meeting of an ISO subcommittee in Warsaw, but none of the qualified people we suggested were able to attend.

We have reviewed the NRC report, *Users' Guides for Radioactivity Standards*, being revised by the subcommittees on radiochemistry and standards. The introduction was prepared here last year.

RADIOISOTOPE SALES

(J. E. Ratledge)

ITEMS OF INTEREST

Orders were received from Measurex Corporation for 1085.1 Ci of enriched krypton-85 and from Donald W. Douglas Laboratories for 200 kCi of promethium-147. Requests for quotations were received from Institute of Nuclear Research, Poland, for a source containing ∿75 kCi of strontium-90; from Ohmart of Canada, Limited, for 105 sources containing ∿200 Ci of cesium-137; and from Saunders-Roe Developments, Limited, England, for 100 kCi of tritium gas.

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

Customer	Isotope	Amount
Large Quantities		
New England Nuclear Corporation	Tritium	7,000 Ci
General Electric Company	Xenon-133	19 Ci
Atomic Energy of Canada, Limited	Cesium-137	25,525 Ci
Minnesota Mining & Manufacturing Company	Cesium-137	500 Ci
Measurex Corporation	Krypton-85, enriched	262.95 Ci
Industrial Nucleonics Corporation	Krypton-85, normal	200 Ci
Withdrawn Items		
International Copper Research Associates	Copper-67	1 lot
Mayo Clinic	Copper-67	2 lots
University of Pittsburgh	Iodine-131	50 mCi
Cleveland Metropolitan General Hospital	Iodine-131	50 mCi
Pacific Northwest Laboratory	Iodine-131	10 mCi
University of Rochester	Iodine-131	100 mCi
Items Used in Cooperative Programs		
University of Southern California	Platinum-195m	∿30 mCi
University of Delhi	Tellurium-127	∿7 mCi
Oak Ridge Associated Universities	Gallium-67	300 mCi
Unusual Items		
University of California, San Francisco	Iron-55	∿60 mCi[a]
Kevex Corporation	Cadmium-109	∿40 mCi[a]
Airco Industrial Gases	Fission product xenon	1,065 liters
Pacific Northwest Laboratory	Fission product xenon	45.1 liters

[a]On loan.

Radioisotope sales proceeds and shipments for the first five months of FY 1972 and FY 1973 are given in Table 2.

Table 2. Radioisotope Sales and Shipments

Item	7-1-71 thru 11-30-71	7-1-72 thru 11-30-72
Inventory items	$148,101	$146,057
Major products	24,247	23,633
Radioisotope services	125,449	60,452
86-Inch Cyclotron irradiations	58,514	37,799
Miscellaneous processed material	30,281	23,198
Packing and shipping	30,705	27,955
Total	$417,297	$319,094
Number of Shipments	1,081	871

ADMINISTRATIVE

Visitors to the IDC are given in Table 3.

Table 3. Visitors to IDC

Visitor	Affiliation	Purpose of Visit
D. B. Duane, W. R. James, Charles Judge, Nate Lang, Ed Maiolatesi, John Rowland, and John Scott	US Army Corps of Engineers, Washington, D.C.	Training program for RIST
Henry Hubble	General Electric Company	Discuss cyclotron operations

PUBLICATIONS

REPORTS

Eugene Lamb, *Isotopic Power Fuels Monthly Status Report for November 1972*, ORNL-TM-4061, Oak Ridge National Laboratory.

A. F. Rupp, *Radioisotope Program (8000) Progress Report for November 1972*, ORNL-TM-4067, Oak Ridge National Laboratory.

A. N. de Gaston, *Removal of Static Electricity from Aircraft Jet Fuel by Irradiation*, MDC-J4192, Douglas Aircraft Company, McDonnell Douglas Corporation, September 1972 (cooperative program using ORNL IDC strontium-90 sources).

Marvin Ben Hertz, *Dosimetric and Flow Analysis of a 100-Ci Strontium-90 Blood Irradiator*, Doctoral Thesis, Vanderbilt University.

INTERNAL DISTRIBUTION

1. M. A. Baker
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59. C. R. Buchanan, AEC, Washington, D. C.
60. R. L. Butenhoff, AEC, Washington, D. C.
61. T. D. Chikalla, PNL, Richland, Washington
62. D. F. Cope, AEC Site Representative, ORNL
63. J. C. Dempsey, AEC, Washington, D. C.
64. B. J. Dropesky, LASL, Los Alamos, New Mexico
65. E. E. Fowler, AEC, Washington, D. C.
66. J. D. Goldstein, AEC, Washington, D. C.
67. A. Gottschalk, Argonne Cancer Research Hospital, Chicago, Illinois
68. F. D. Haines, AEC, Washington, D. C.
69. W. D. Holloman, AEC, Washington, D. C.
70-72. J. H. Jarrett, PNL, Richland, Washington
73. D. K. Jones, AEC, Richland, Washington
74. J. Lawrence, Lawrence Berkeley Laboratory, Berkeley, California
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83. W. D. Sandberg, AEC, Aiken, South Carolina
84. R. W. Shivers, AEC, Washington, D. C.
85. F. J. Skozen (Krizek), Argonne Cancer Research Hospital, Chicago
86. Robert E. Smith, Atlantic Richfield Hanford Company, Richland, WA
87. L. G. Stang, Jr., BNL, New York
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