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ISOTOPE PROGRAM (NT) PROGRESS REPORT FOR QUARTER ENDING DECEMBER 31, 1973

J. H. Gillette

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ISOTOPES DEVELOPMENT CENTER

Isotopes Division

ISOTOPE PROGRAM (NT) PROGRESS REPORT FOR QUARTER ENDING DECEMBER 31, 1973

J. H. Gillette

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FEBRUARY 1974

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ISOTOPE PROGRAM (NT) PROGRESS REPORT FOR QUARTER ENDING DECEMBER 31, 1973

TARGET FABRICATION AND DEVELOPMENT

General Technology

Many new types of samples were produced during this quarter which were requested by other AEC laboratories for research in low-, medium-, and high-energy physics. A general listing of unusual samples heretofore not produced by the Isotope Target Laboratory (ITL) is given in Table 1 for reference.

Table 1. New Sample Preparations

Element	Film Thickness, mg/cm^2	Substrate	Remarks
Mn	19, 9, 4	None	Brittle; vapor-deposited on Al, latter dissolved in caustic
Bi	35, 12	None	Vapor-deposited on detergent plus NaCl, parted on water; brittle, but large-area targets possible
Se	35, 12	Mylar	Vapor-deposited; tendency to undergo phase change
Cs_2O	0.1	C: 50 $\mu g/cm^2$	Vapor-deposited on self-supported carbon film; must be kept in Ar atmosphere
Be	0.05	None	Vapor-deposited on detergent plus NaCl, parted on water, mounted on grids
Sb	35	Ni: 220 mg/cm^2	Vapor-deposited; bright, adherent coating; efficiency ~25%
Cr	50	None	Electroplated; heaviest known isotopic deposit; stripped from Al substrate by dissolution of latter in caustic

In addition to the variety of new samples of unusual thicknesses noted in Table 1, technologies were developed to produce bulk samples especially useful in high-energy physics at the LAMPF. In particular, ^6Li and ^7Li metal was cast into 5-cm-dia cylinders of various lengths

in such a way as to include little or no oxide and nitride contaminants; the metal was cast to within 0.1 mm of the required diameter in an argon-atmosphere glove box. Cast samples were carefully cut to correct volume and then pressure—fitted at ∿8000 psig to precision-machined containment vessels (aluminum shell). By this technique, the exposed parallel faces were finished in a polished condition and were plane-parallel to ±0.013 mm. Finally, protective plastic windows were fitted and sealed over the cylinder ends with epoxy resin. Pressure die construction and casting development proved extremely important to the successful fabrication of these samples.

Lithium metal isotope wire was produced in the same facility as was the metal casting previously described. From a brass extrusion die having a hardened tool-steel orifice, 0.5-mm dia wire was formed at a temperature of ∿120°C, which permitted plastic flow of the metal under pressures of only a few hundred pounds per square inch. Clean, uniform-diameter wire (∿50 ft) was produced which was then cut into uniform lengths and sealed in high-purity vanadium for fast reactor dosimeter measurements. In this case, the helium formed by ^{6}Li fission was analyzed using a supersensitive helium mass spectrometer at Atomics International to determine neutron fluence at the dosimetry points in the EBR II Reactor.

Reduction--Distillation of Pu and Am Isotopes

Reduction--distillation of lanthanide oxides to produce ultra-high-purity isotope metals has been used successfully over the past six years, but has been limited to americium and curium -- largely as the result of inadequate compatibility of other actinide metals with the tantalum metal reaction vessel and distillation "column" used in this process. However, thermodynamic considerations indicated that neptunium and plutonium metals could well be produced from their respective oxides by reduction--distillation if a reaction vessel could be found which would be "opaque" to metal vapors and inert to liquid metal produced in the process.

To solve the compatibility problem, tantalum carbide crucibles of appropriate dimensions were obtained and the reaction of PuO_2 with the metal as the reductant was studied experimentally. A quantity of 3.5 g $^{239}PuO_2$ (88.19%) was reduced using 4.5 g of thorium. A pressed pellet of this mixture was reacted at 1750°C, but only 1.8 g of product Pu was obtained. At a temperature of 1900°C (45-min heating period), an additional 1.2 g of metal was collected for a total reduction--collection efficiency of ∿97% -- a remarkable efficiency even for more easily reducible materials such as calcium. Upon analysis by spark-source mass spectrometry, the impurities listed in Table 2 were observed.

The more volatile impurities, e.g., Ba, Ca, Pb, and Sn, can be eliminated by redistillation and rejecting the first 5-10 mg collected. The thorium impurity can be easily removed by redistillation of the plutonium; silicon impurity originates from the quartz collector and can be better removed by more careful abrasive cleaning of the metal surface nearest the

quartz. Generally, as with the lanthanides, plutonium metal can be formed in quantities from 0.5 g to multihundred grams by this process with subsequent total impurity content of <300 ppm, including oxygen and nitrogen. This process permits the conversion of small quantities of isotope oxides, which is otherwise infeasible because the rarity and monetary value preclude "bomb reduction."

Table 2. Spark-Spectrographic Analysis of ^{239}Pu Metal

Element	Weight, ppm	Element	Weight, ppm
Al	5	Sn	20
B	0.2	Sr	4
Ba	600	Ta	0.7
Ca	5	Th	3000
Cr	20	Ti	4
Cu	4	V	0.2
Fe	70	Zn	1
K	5	^{239}Pu	M
Mg	1	^{237}Np	10
Mn	0.7	Cl	70
Na	3	S	10
Ni	30	La	6
P	0.4	Ce	6
Pb	1500	Pr	0.2
Si	2000	Nd	2

In the near future, 20 g of ^{237}Np metal will be produced by the reduction--distillation method. Past experience with the lanthanides and actinides would indicate that neptunium metal formed by this process may have considerably different physical properties (e.g., ductility) than bomb-reduced material, which normally contains large concentrations of impurities, especially oxygen and fluorine.

Twenty grams of ^{243}Am metal was produced by the reduction--distillation process for crystallographic and metallurgical studies at ANL and ORNL. This amount of material was the largest quantity ever produced by the ITL.

STABLE ISOTOPE SEPARATIONS

Isotope separations of Cd, Os, Pd, and Zr progressed throughout the 3-month interval in a total of 18 electromagnetic isotope separators. A palladium isotope separation, initiated at the beginning of the quarter to provide up to 100 g of each isotope for the Research Materials Collection (RMC), is under way in two beta-type separators; osmium is being separated and collected in two alpha-type (48-in. radius) calutrons; cadmium is in a segment of eight beta-type separators; and zirconium is being enriched in six separators modified with 255° inhomogeneous equipment. The osmium and palladium isotope separations are being conducted on an 8-hr, 5-day-week basis, while cadmium and zirconium separations are being made on a 24-hr, 5-day-week basis.

The recently developed technique of reacting appropriate platinum metals directly in the arc chamber of the calutron ion source with ClF_3 has been extended to the osmium and palladium separations with improved results. Palladium, for example, is being collected at ∿6 times the rate of the best previous separation, while osmium isotopes are being collected ∿3 times as rapidly as in the only previous separation. Preliminary mass analyses of collected samples from both separations are consistently higher than obtained previously.

Comparative Assay Data

Palladium			Osmium		
Isotope	Current Separation, %	Series NO (1967), %	Isotope	Current Separation, %	Series JS (1960), %
102	84.6	75.45	184	5.0	2.25
104	96.7	89.75	186	76.4	61.27
105	98.2	94.51	187	70.2	45.76
106	98.1	96.66	188	94.2	87.7
108	99.2	98.11	189	95.1	87.3
110	98.3	96.98	190	97.5	95.46
			192	99.3	98.68

Although there is a distinct improvement in the separation rates for osmium and palladium through the use of the new charge system, the change does not equal the dramatic improvement achieved earlier[1] with iridium and platinum. This is explainable, however, since the relative advantage of ClF_3 is not as great: i.e., osmium can be separated using volatile osmium tetroxide, and palladium has the highest vapor pressure of the platinum metals and can be vaporized without the need for electron-bombardment heating.

A short second-pass separation of ^{115}Sn was completed in one of the spare beta-type calutrons during the quarter. The charge material for this separation consisted of ∿4.7 g of tin (as SnS_2) pre-enriched to 18.34% in ^{115}Sn. The recovered product, 39 mg of 97.33% ^{115}Sn, will be used in coulomb-excitation and other physics-research studies.

[1] J. H. Gillette, *Isotope Program (5000) Progress Report for Quarter Ending June 30, 1973*, ORNL-TM-4338, p. 5, Oak Ridge National Laboratory.

Source Development

Development activities in the stable isotopes program this quarter were concerned with (1) varying electrode structures as a means of improving isotope separations (e.g., hafnium), in such areas as increasing ion output and controlling sparking; (2) ion-source modifications to improve the purity of tellurium isotopes (specifically ^{122}Te); and (3) continuing the development of the 180° sector isotope separator computer-control system as it relates to extending the system to a calutron operation.

Small-diameter electrodes made from 3/32-in.-dia tungsten rods were used first in high-purity silicon separations. Since then their use has been extended to scheduled separations of rubidium and cadmium, and to developmental runs involving separation of tellurium and hafnium. These electrodes, supporting other source innovations, contributed to the separation of 99% ^{122}Te (previous best, 95.44%) and of >95% ^{179}Hf (previous best, 86.98%) containing only 1.62% ^{180}Hf. Neither of the latter two efforts is complete. Hafnium work is hampered by growth of hafnium on the ionization filament and excessive sparking in the electrode region -- independent of the electrode type in use. This condition is undoubtedly augmented by excessive pressure in the slit region as unreacted CCl_4 escapes through the ion-exit slit. With HfO_2 used as an initial feed compound that reacts with CCl_4 internally within the ion source to form a volatile feed, approximately ten times the needed stoichiometric amount of CCl_4 is introduced to produce the necessary feed. This excess can be reduced by 15-20% by elevation of the temperature of the reaction region; however, this approach elevates the temperature of the ion-exit slit, and this in itself aggravates the sparking. Improvement in the completeness of the $HfO_2 + CCl_4$ reaction must be achieved before appreciable improvement in this separation can be realized.

RADIOISOTOPE SEPARATIONS

Plutonium-241

A plutonium isotope separation (Series bb), which was started July 9, 1973 in two of the alpha-contained calutrons with the major objective of adding multigram quantities of ^{241}Pu to the isotopes research pool, was terminated November 27, 1973, after accumulating the following estimated quantities of isotopes:

Isotope	Estimated Recovery* (∿g)	Estimated Purity (∿%)
240	85	98
241	25	>90
242	25	>80

*Product of accumulated monitored ion currents and probable collector retention.

The starting material for this separation consisted of ∿733 g of plutonium (as PuO_2), obtained by blending a batch of unused material with ∿165 g of plutonium recovered from a previous separation. The average approximate isotopic composition of the charge material was ^{238}Pu, 0.92%; ^{239}Pu, 23.12%; ^{240}Pu, 45.20%; ^{241}Pu, 13.91%; ^{242}Pu, 16.83%; and ^{244}Pu, 0.015%. In all, ∿1200 g of plutonium was vaporized (by an in-situ chlorination with CCl_4) into the calutrons by including material recovered through in-process recovery--recycle operations.

Overall, the separation consisted of 26 separate runs that resulted in 2324 hours of ion-beam reception.

High-Purity Plutonium-241

A special high-purity ^{241}Pu separation was completed in one of the alpha-contained separators with the objective of obtaining 1 g (or more) of ≅99% ^{241}Pu. The charge material for this separation consisted of 30.4 g of plutonium (as PuO_2) obtained by blending a sample returned from Idaho Nuclear Corp. with two of the lower-purity samples recovered from the previously described plutonium separation. The approximate isotopic composition of the blended charge material was ^{238}Pu, 0.58%; ^{239}Pu, 47.10%; ^{240}Pu, 21.84%; ^{241}Pu, 23.11%; and ^{242}Pu, 7.36%.

The separation, which consumed 60 ion-beam hours, yielded ∿1.2 g of ≧98% ^{241}Pu and ∿0.3 g of >90% ^{242}Pu. Chemical recovery of these enriched products was still incomplete at the end of the quarter.

Beryllium-10 Enrichment

Another[2] special ^{10}Be enrichment program is currently under way in three of the singly contained calutrons in order to obtain a few milligrams of highly enhanced ^{10}Be for research use. The charge material for this separation, which now consists of ∿70.4 g beryllium (as "low-fired" BeO), was obtained by reclaiming the unresolved charge material from calutron components used in the earlier enrichment effort.[3] It is presumed that the ^{10}Be content of the recovered material is still ∿700 ppm (the material was not reassayed).

180° OAK RIDGE SECTOR SEPARATOR DEVELOPMENT

Funds were approved in November 1973 for the modification of an existing calutron in order to develop a computer-controlled system. Approximately 50% of the $15,000 approved for this project has now been committed for the purchase of equipment which will interface the ORSIS PDP-8/I computer to an R&D calutron for the purpose of data acquisition and control. However, delivery on all items will not be completed before July 1974. The

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[2] *Ibid*, p. 7.

[3] J. H. Gillette, *Isotope Program (NT) Progress Report for Quarter Ending September 30, 1973*, ORNL-TM-4410, p. 6, Oak Ridge National Laboratory.

hardware installation on the R&D calutron will be completed by August 1974 and will be ready for testing computer control algorithms during actual operation. Approximately 50% of the calutron separator equipment modifications have been completed.

Funds were also approved in 1973 for the update of the ORSIS computer-control system to provide improved ORSIS control and to enable this system to control an R&D calutron. All equipment has been ordered and deliveries should be completed during March 1974. This system will employ a real-time software monitor that will utilize 32K of core memory, 1.5M words of disk memory, and the ORSIS PDP-8/I computer for real-time control and operator--system interaction. The updated computer-control system should be completed and ready for applications programming for both the ORSIS and the R&D calutron by June 1974.

The calutron automation proposal has been revised; it now incorporates new cost-benefit data and several new control features which are thought to be feasible as a result of studies on the ORSIS control system. This proposal continues to show a high benefit/cost ratio and suggests that the more efficient system that would result may actually be necessary in order to remain competitive with foreign suppliers (principally Russian).

Rewiring and reworking of the sector control system have been going on for most of this quarter. The rewiring is needed to comply with OSHA standards and to reduce noise pickup on signal lines by placing all wiring in conduit. The reworking is necessary in order to permit interaction with the updated computer-control system, to provide better ion-source arc control, to permit higher ion-source arc voltages for the generation of multiply charged ion beams, and to provide additional vacuum-system interlocks. This revision is now 90% complete.

Several requests for high-purity isotope separations are now pending; these could be performed in a single pass on the ORSIS from normal-abundance charge material. (The calutron would require two or three passes to accomplish the same task.) For example, ^{156}Dy will now be used to produce ^{157}Dy which is being investigated as a tumor-imaging agent in humans. However, the 0.0524% ^{156}Dy starting material must be enriched to >99% -- a feat which has already been performed on the ORSIS in a single pass. Similar requests have also been received for sizeable quantities of high-purity ^{205}Pb, ^{122}Te, and ^{96}Ru. Requests of this type are becoming more frequent -- a fact that has prompted a study of ways to increase the efficiency of production of these materials. Theoretical studies based on currently achievable ion-beam intensities with the first ORSIS indicate that the second ORSIS might increase ion-beam intensities from the current level of 1/10 up to 1/3 to 2/3 those of the calutron for most elements. Approximately 50% of this increase would result from an increase in the size of the beam-transmission system, with the rest coming principally from a redesign of the magnetic focusing system. Studies are under way to determine how much of this increase might be practially achievable through modification of the first ORSIS.

The ORSIS has also been modified to produce higher intensities of multiply charged ions for use in ion doping and in the collection of gaseous elements in solid substrates. No data have yet been accumulated, but ion energies up to 500 keV at practical intensities appear to be feasible.

The decel method of collecting ion beams at a few hundred electron volts has proved to be very powerful in preparing very high purity, thin, accelerator targets and very high-purity bulk samples. However, it is not useful for collecting high-purity mercury or for collecting high-purity gaseous isotopes by ion implantation, since high-energy ion beams are required to make the collection effective. A new system is being studied which would energy-filter the ion beam and then reaccelerate it for final implantation or collection. This system should make very high-purity gaseous-isotope implants possible.

CHEMICAL PROCESSING

Material in Process

Elements in chemical processing during the quarter ending December 31, 1973, are Cd, Hf, Ir, Hg, Os, Pd, Pt, Rb, Zn, and Zr.

Material Added to Inventory

Isotopic material released or made available to the Sales Department during the fourth quarter of calendar year 1973 is shown in Table 3, along with the appropriate separation data.

In addition to the 10 new lots of isotopes released to inventory, 15 loan-return samples were processed and returned to inventory.

ISOTOPES PREPARATIONS AND SALES

The stable isotope and radioisotope target and research material preparations and the isotopes sales and services for the quarter ending December 31, 1973, are given in Tables 4, 5, and 6, respectively.

Table 3. Operations Status -- October--December 1973

Element and Isotope	Series	Run Start	Run Finish	Innage Hours	Tank Hours	Charge Material	Estimated Recovery (g)	Estimated Purity (%)	Actual Recovery (g)	Actual Purity (%)	Requirements
Ir-191	PM	1-26-73	9-28-73	1,912	2,600	Ir + ClF_3			53.926 2.412	98.17 97.13	50 g >90
Ir-193	PM	1-26-73	9-28-73	1,912	2,600	Ir + ClF_3	90	>99			50 g >95
Hg-196	PG	3-13-72	6-1-73	26,816	32,736	Hg			1.219 0.397 0.264	25.18 19.28 13.94	50 g ∿47
Pt-194	PI	4-3-72	5-16-73	3,000	4,400	Pt + ClF_3			58.080	97.41	50 g >90
Pt-195	PI	4-3-72	5-16-73	3,000	4,400	Pt + ClF_3			59.831	97.28	50 g 47-60
Rb-85	PP	6-4-73	9-28-73	13,744	16,128	Rb_2CO_3	450	>98			7.3 g >99
Rb-87	PP	6-4-73	9-28-73	13,744	16,128	Rb_2CO_3	215	∿98			140 g >98
Sn-115[a]	R&D	3-1-73	10-17-73	80	-	SnS_2 + H_2S			0.013[b]	97.33	0.1-0.15 g 95
Zn-70	PL	1-2-73	4-27-73	13,309	15,744	Zn			3.496 0.962	79.59 64.64	3 g ≥90

[a]Second-pass separation for Division of Physical Research.

[b]Partial shipment.

Table 4. Stable Isotope Preparations

Isotope	Number of Preparations	Isotope	Number of Preparations
Antimony-121	1	Molybdenum-92	3
Antimony-123	1	Molybdenum-94	1
Normal Beryllium	16	Molybdenum-100	1
Boron-10	1	Neodymium-142	1
Boron-11	1	Neodymium-146	1
Cadmium-110	1	Neodymium-150	1
Cadmium-114	1	Nickel-58	1
Calcium-44	2	Nickel-59	1
Normal Chromium	18	Nickel-61	1
Chromium-52	1	Palladium-110	2
Normal Cobalt	2	Platinum-196	1
Copper-63	3	Normal Praseodymium	3
Erbium-167	1	Samarium-151	1
Erbium-170	1	Samarium-152	1
Gadolinium-157	4	Silicon-30	1
Hafnium-177	1	Silver-107	1
Hafnium-179	2	Normal Tantalum	1
Iron-57	3	Titanium-48	5
Lead-204	1	Tin-116	2
Lead-206	2	Tin-120	1
Lead-208	3	Tin-122	1
Lithium-6	9	Ytterbium-168	1
Lithium-7	15	Ytterbium-174	2
Magnesium-24	2	Ytterbium-176	1
Normal Manganese	100	Zinc-68	1
		Zirconium-90	4
		Zirconium-94	1

Table 5. Radioisotope Preparations

Isotope	Number of Preparations	Isotope	Number of Preparations
Berkelium-249	1	Scandium-45	5
Carbon-14	2	Thorium-232	2
Curium-244	1	Uranium-235	2
Neptunium-237	2	Uranium-236	2
Plutonium-242	1	Uranium-238	4

Table 6. Isotopes Sales and Services

	Shipments	Revenue
EM Isotopes Sales	652	$357,192
Non-EM Isotopes Sales	108	19,726
Services		
Stable Isotope Target Preparations		31,091
Radioisotope Target Preparations		4,300
Special Services		5,275
Reprocessing Returned Loans		6,580
Work for Others		3,490
Total Services		50,736
TOTAL	760	$427,654

ADMINISTRATIVE

Visitors to the IDC are shown below.

Visitor	Affiliation	Subject Discussed
Dr. G. J. Wasserburg and D. Papanastassiou	Div. of Geological and Planetary Sciences Cal. Inst. of Technology Pasadena, CA	Isotopic enrichment of lead-205

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