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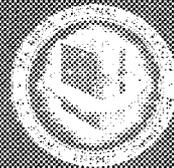
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Isotope Program (NT) Progress Report  
for Quarter Ending December 31, 1974

J. H. Gillette



**OAK RIDGE NATIONAL LABORATORY**

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ISOTOPES DEVELOPMENT CENTER

Isotopes Division

ISOTOPE PROGRAM (NT) PROGRESS REPORT  
FOR QUARTER ENDING DECEMBER 31, 1974

J. H. Gillette

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OAK RIDGE NATIONAL LABORATORY  
Oak Ridge, Tennessee 37830  
operated by  
UNION CARBIDE CORPORATION  
for the  
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

ISOTOPE PROGRAM (NT) PROGRESS REPORT  
FOR QUARTER ENDING DECEMBER 31, 1974

STABLE ISOTOPE SEPARATIONS

At the end of the quarter, isotope separations of Se, Te, and Sn were in progress in 14\* electromagnetic separators. The selenium isotope separation was initiated early in November 1974 in an 8-tank segment of beta-type separators to recover 1.5 to 2 g of >75%  $^{74}\text{Se}$  for the Sales inventory. Selenium-74 is the precursor of  $^{75}\text{Se}$  which is used in nuclear medicine as a diagnostic scanning agent. Tellurium and tin isotopes, which are also used in the preparation of medically useful radioisotopes, are being separated in 3 tanks each of 255° modified isotope separators. All separations are being made on an 8-hr, 5-day-week basis.

During the quarter, in order to utilize available separators and manpower for the above-mentioned selenium separation, a calcium isotope separation (intended to recover ~10 g of >97%  $^{48}\text{Ca}$ ) was terminated after obtaining ~25% of the suggested requirement. In addition, the palladium and ruthenium separations (which were being made to provide priority requirements for the RMC) were also terminated short of stated requirements. These separations were concluded due to the closing of Bldg. 9731.

As in other recent platinum-metal (Os, Ir, Pt) isotope separations, the use of  $\text{ClF}_3$  in vaporizing and transporting the ruthenium and palladium metals in the ion source dramatically improved several separation parameters over earlier separations, as shown in Tables 1 and 2. The marked improvement in isotopic purity did not occur with ruthenium, since the previous separation was made in 255° units using previously prepared  $\text{RuF}_5$  charge.

DEVELOPMENT ACTIVITIES

Development activities during the quarter have been concerned with (1) improving process efficiency (P.E.) through ion-source modification in connection with a special separation of reactor-produced  $^{41}\text{Ca}$  and a second-pass separation of  $^{124}\text{Te}$ , (2) investigating (in process) the feasibility of a two-arc system for selenium, (3) evaluating  $\text{SnTe}$  as a charge material, and work with the sector separator which included (4) continuing efforts to modify the sector machine and an existing R&D calutron with computer controls and (5) determining sources of contamination in the sector separator decel receiver.

Ion Source Development

The need for attaining the highest possible P.E. in a scheduled  $^{41}\text{Ca}$  separation led to the insertion of a smaller arc chamber liner into the

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\*Separations in Bldg. 9731 were terminated during the quarter and personnel moved to Bldg. 9204-3.

Table 1. Comparison of Ruthenium Isotope Collections

|  | Series MH <sup>a</sup><br>(1964) | Series PU<br>(1974) |
|--|----------------------------------|---------------------|
| Percent innage                           | 58.7                             | 66.0                |
| Avg. total ion output (mA)               | 7.3                              | 22.45               |
| Process efficiency (%)                   | 3.0                              | 6.0                 |
| Mass assays:                             |                                  |                     |
| <sup>96</sup> Ru                         | 98.07                            | >97 <sup>b</sup>    |
| <sup>98</sup> Ru                         | 89.00                            | >85 <sup>b</sup>    |
| <sup>99</sup> Ru                         | 98.8                             | >95 <sup>b</sup>    |
| <sup>100</sup> Ru                        | 97.24                            | >95 <sup>b</sup>    |
| <sup>101</sup> Ru                        | 97.73                            | >97 <sup>b</sup>    |
| <sup>102</sup> Ru                        | 99.53                            | ~99 <sup>b</sup>    |
| <sup>104</sup> Ru                        | 99.7                             | ~99 <sup>b</sup>    |
| Charge material                          | RuF <sub>5</sub> <sup>c</sup>    | Ru+ClF <sub>3</sub> |
| Charge consumption rate (g Ru/innage hr) | 0.9                              | 1.4                 |
| Total Ru monitored (g)                   | 38.564                           | 49.637              |
| Total tank hours                         | 2395                             | 888                 |

<sup>a</sup>255°-modified isotope separators.

<sup>b</sup>Estimates based on preliminary-type mass assays.

<sup>c</sup>Externally prepared at ORGDP.

Table 2. Comparison of Palladium Isotope Collections

|  | Series NO<br>(1967) | Series PR<br>(1974) |
|--|---------------------|---------------------|
| Percent innage                           | 64.7                | 67.8                |
| Avg. total ion output (mA)               | 2.6                 | 22.6                |
| Process efficiency (%)                   | 7.0                 | 4.7                 |
| Mass assays:                             |                     |                     |
| <sup>102</sup> Pd                        | 75.45               | >75 <sup>a</sup>    |
| <sup>104</sup> Pd                        | 89.75               | 95 <sup>a</sup>     |
| <sup>105</sup> Pd                        | 94.51               | >95 <sup>a</sup>    |
| <sup>106</sup> Pd                        | 96.66               | >98 <sup>a</sup>    |
| <sup>108</sup> Pd                        | 98.11               | >98 <sup>a</sup>    |
| <sup>110</sup> Pd                        | 96.98               | >98 <sup>a</sup>    |
| Charge material                          | Pd <sup>b</sup>     | Pd+ClF <sub>3</sub> |
| Charge consumption rate (g Pd/innage hr) | 0.15                | 1.9                 |
| Total Pd monitored (g)                   | 68.0                | 287.8               |
| Total tank hours                         | 9856                | 4708                |

<sup>a</sup>Estimates based on preliminary-type mass assays.

<sup>b</sup>High-temperature ion source (not electron bombardment).

conventional arc chamber of the high-temperature ion source.<sup>1</sup> Used in conjunction with a cylindrical charge container and smaller ion-exit slit (1/8 in. x 1-3/4 in.), both made of stainless steel, this source yielded 27.8% P.E. when charged with 6.0 g of calcium (as CaO) compressed into pellets with 24.0 g of metallic lanthanum. This is an appreciable improvement (factor of 2 to 3) over results from standard ion sources charged with similar quantities of CaO and lanthanum. Ion output equalled that of previous efforts with small charges but was only ~25% that achieved from 100-g charges of metallic calcium.

By modifying the heaters slightly to reduce power input, the above ion source was also tested with 12-g charges of elemental tellurium. The P.E. averaged 16% (prior efforts yielded 4 to 8%). Ion outputs equalled prior ones; assays of the <sup>122</sup>Te show equal performance in both cases. (The peak-to-valley ratios used as in-process guides indicated that focus was inferior during operation, yielding the high values of P.E.) Filament life was definitely reduced and only 5 to 6 g of tellurium could be vaporized prior to filament failure. The results with both calcium and tellurium show this unit to be superior to any other now available and its use in the separations of <sup>41</sup>Ca and second-pass <sup>124</sup>Te is planned.

In pursuing the work on improved process efficiency, it was realized that considerable loss in feed occurs during the latter stage of a run cycle when charge vapor pressure is elevated in an effort to sustain ion output at its previous level. Various conditions or factors (charge deposits, filament wear, insulating layers, etc.) have been associated with the reduction in ion output with the aging of a run, but in short runs with very little feed material it seems that filament wear must be a major contributing factor. If so, the contribution can be explained as follows: The filament feeds energetic electrons through a slot (usually 1/8 in. x 1/2 in.) into the primary arc column. The primary arc is positioned directly behind an ion-extraction slit (usually 3/16 in. high, thus leaving 5/32 in. of the primary arc hidden above and below the slit aperture). During wear, the filament becomes thinner in the central region of the slot and the portions of the arc hidden behind the upper and lower defining edges of the slit receive relatively fewer electrons.

In order to determine how much the hidden portions of the primary arc column contribute to the total ion output of a source, an ion source was fitted with two filaments and two electron collimating slots, one above and the other below the slot opening; thus the region directly back of the ion-exit slit was devoid of electrons supplied by a filament. Operating independently, the top arc yielded ~40 mA, and the bottom arc ~20 mA of Ca<sup>+</sup>, and both simultaneously >100 mA, with a maximum of 140 mA. Simultaneously, the two arcs hidden from the extraction slit yielded an ion output equal to, or even exceeding, that normally drawn from the standard arc geometry.

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<sup>1</sup>J. H. Gillette, *Isotope Program (NT) Progress Report for Quarter Ending Sept. 30, 1974*, ORNL-TM-4745 (Dec. 1974).

### In-Process Selenium Two-Arc Experiment

A two-arc experiment is being carried out in one of the eight selenium separation units in order to evaluate the possibility of increasing the total ion output without materially affecting the isotopic purity of  $^{74}\text{Se}$  (currently the "wanted" isotope and the one that will bear most, or all, of the accumulated cost of the separation). Physically, the two-arc equipment for selenium is very similar to that described for calcium in the last quarterly report, except that the collectors are aluminum rather than graphite and/or copper.

After a two-month interval ( $\sim 700$  innage hours), the total ion output of the combined arcs averaged 16.4 mA, based on monitored ion currents to the  $^{82}\text{Se}$  pocket. This compares to an average of 10.8 mA\* in the seven adjacent single-arc collections during roughly the same time interval.

To date, two  $^{74}\text{Se}$  samples have been recovered, one from each arc, and preliminary mass analyses of representative portions show 60.5%  $^{74}\text{Se}$  from the top receiver and 55.2%  $^{74}\text{Se}$  from the bottom arc -- low when compared to the average obtained in the last series (78.61%  $^{74}\text{Se}$ ); but the values are equal to or better than those obtained in prior separations (a high of 59.14%). The output of the two-arc experiment is  $\sim 52\%$  more than that of the adjacent single-arc runs and, although the  $^{74}\text{Se}$  abundance is somewhat lower (55-60% compared to 78%), the experiment is still considered a success since there is  $\sim 15\%$  more contained  $^{74}\text{Se}$  in the double-arc product than in the single-arc product.

### Evaluation of SnTe as a Charge Material

As reported earlier,<sup>1</sup> SnTe appears to be at least as effective as elemental tellurium as a charge, using ion output and peak-to-valley ratios as criteria for judgement. However, erosion of aluminum collectors used in simultaneous collection of isobaric isotopes and erratic isotopic enrichment have been observed. The latter problem (enrichment) is attributable to rejection of material from the aluminum collectors (which have also complicated the chemical separation and refinement procedures). Thus, really satisfactory usage of SnTe feed rests in alteration of receiver designs and pocket materials.

### 180° Oak Ridge Sector Separator Development

Development efforts are continuing in order to provide the capability with ORSIS to prepare highly enriched isotopic targets directly for all isotopes without using pre-enriched charge. Efforts are also being made to provide high-purity material, in situ, in the directly prepared targets so as to avoid the chemical contamination that often results otherwise from plating, evaporation, and chemistry procedures.

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\*Series PH (1972) also used CdSe and averaged 10.7 mA for  $\sim 19,000$  innage hours.

<sup>1</sup>*Op. cit.*

Thin targets of  $^{154}\text{Gd}$  at  $25 \mu\text{g}/\text{cm}^2$  on  $80 \mu\text{g}/\text{cm}^2$  carbon foil have been prepared at 175 eV deposition energy for coulomb-excitation studies. The 99.7% (estimated) isotopic purity of the target material was obtained from natural-isotopic-abundance charge material. In contrast, the best  $^{154}\text{Gd}$  second-pass product obtained in the calutron is 99.35%.

#### Computer Control of Calutrons

Instrumentation and Controls Division support was resumed during December 1974 in order to complete the interfacing of new peripheral equipment to the ORSIS computer and to complete and test the new ORNL-designed real-time computer monitor system. This monitor system will permit any combination of tasks to be executed, which are written in either modified Digital Equipment Corporation FOCAL or assembly language. All tasks can be both interrupt driven by the process and scheduled. This work should be finished during the first quarter of 1975 so that applications programming for both the ORSIS and R&D calutron can begin during the second quarter, utilizing the new system.

#### Sources of Contamination in the Decel Receiver

Several experimental ORSIS separations of dysprosium isotopes have been completed in order to determine the most significant contamination mechanisms which exist in current separations and also to determine what changes are needed in equipment and techniques to significantly exceed the current best single-pass enrichments of 400,000 (from normal abundance of 0.0524% to 99.53%) with  $^{156}\text{Dy}$ . The primary mechanism involved in the decel equipment and method of isotope collection is the deceleration of the isotope beam (e.g.,  $^{156}\text{Dy}$ ) from 40,000 eV to 300 eV (i.e., ions with less than 39,700 eV are not accepted). The primary second-order contamination mechanism then appears to be caused by neutral ions.

Dysprosium-156 was collected for 35.0 hr at +300 eV in the decel collector; then the beam energy was lowered so as to put the  $^{162}\text{Dy}$  directly into the grounded  $^{156}\text{Dy}$  collection slot for 4.5 hr. The  $^{162}\text{Dy}$  primary ions had an energy deficit of  $\sim 1100$  eV and no  $^{162}\text{Dy}$  current was monitored. The  $^{156}\text{Dy}$  assay was normal and would have been  $\sim 99\%$  except that the  $^{162}\text{Dy}$  content was raised to 61% instead of a normal 0.2%. Without the decel, 1.57%  $^{156}\text{Dy}$  would have resulted instead of the 38% actually achieved in this experiment. The decel permitted only  $\sim 2.6\%$  of the  $^{162}\text{Dy}$  to contaminate the sample. These extra  $^{162}\text{Dy}$  atoms could have gotten into the sample only by first neutralizing on the entrance slits to the collector and then proceeding unheeded into the collector. These results have prompted the following action: (1) the mechanical shutter in front of the collection slot is closed by computer control whenever the mass or ion energy is out of tolerance, (2) the computer closes the shutter whenever the accelerating voltage is being recycled (sweeps all beams over the collection slot), and (3) the shutter is closed during periods of rapidly accelerating voltage breakdown. An experiment will be conducted in the near future in which the partially analyzed energy beam will be deflected out of this neutral flux to attempt to move the enrichment factor into the millions range for a single pass. One important thing about these results is that

they are obtained with a compact device -- i.e., not a full second-stage magnet and/or electrostatic analyzer but a more complicated version of receiver.

A  $^{163}\text{Dy}$  separation has been completed in order to determine if the excellent decel collection results with  $^{156}\text{Dy}$  (all other Dy masses heavier) can be expected in other elemental separations where some or all contaminating masses are lighter. The resulting single-pass sample assayed 99.87%  $^{163}\text{Dy}$ \* and had contaminating factors from other masses which were consistent with the  $^{156}\text{Dy}$  results. The decel technique and equipment thus appear to be applicable to all isotopic separations with ORSIS.

The following developments to the separator and collector should be pursued in order to fully exploit this technique: (1) construction of a new beam-flight-tube assembly and supporting structure for the receiver region to increase collection rates to  $\sim 1/2$  those of the calutron at these high enrichments, (2) extension of this technique to collection of more than one isotope simultaneously, (3) ion optical design on the decel to increase enrichment factors for those isotopes needed, and (4) better separator-computer interface hardware for the increased quality control demanded at these enrichment levels. In addition, this added hardware would permit the computer to minimize errors in operational sequencing which now occur with manual control.

## RADIOISOTOPE SEPARATIONS

### Thorium-230

A  $^{230}\text{Th}$  isotopic enrichment program, which was initiated in one of the doubly contained electromagnetic isotope separators in January 1974, was concluded the middle of October 1974 upon collecting  $\geq 10$  g of  $\sim 90\%$   $^{230}\text{Th}$ . Following termination of the separation process, three of the 16 preliminary samples which had the highest isotopic purity were blended and chemically refined to provide 3.079 g of 91.54%  $^{230}\text{Th}$  for the Heavy-Element Research Pool. The remaining 13 preliminary samples, along with two inventory lots containing 2.276 g Th, were blended and chemically refined as lot FTh-6 to provide 14.955 g of 89.13% feed material for a second-pass separation. This is scheduled for early 1975 and is expected to yield  $\geq 1$  g of 99.9%  $^{230}\text{Th}$ .

### Thorium-232

A second thorium separation (using "normal" feed from which the  $^{228}\text{Th}$  decay product was to be removed because of its interference in cross section measurements) was started in September 1974 in the four singly contained separators normally allocated for uranium separations and was completed in early October with the collection of  $>100$  g of  $^{232}\text{Th}$ .

A "before" radiometric analysis performed on the feed material by J. F. Emery, Analytical Chemistry Division, showed  $1.18 \times 10^{-10}$  g  $^{228}\text{Th}/\text{g}$   $^{232}\text{Th}$ . Radiometric analyses on the two product samples showed  $3.98 \times 10^{-13}$  g  $^{228}\text{Th}/\text{g}$   $^{232}\text{Th}$  and  $4.69 \times 10^{-13}$  g  $^{228}\text{Th}/\text{g}$   $^{232}\text{Th}$  (or depletion factors of

\*This assay may be compared to  $^{163}\text{Dy}$  assays of  $\sim 90\%$  for the calutron and  $\sim 97\%$  for the 255° separator.

296 and 252, respectively). The two samples consisted of 20.3 g and 82.0 g of contained thorium, respectively.

Following initial recovery of the individual samples from their collector pockets, the two blends were made; these were dissolved in strong nitric acid to obtain 7M HNO<sub>3</sub> solutions and then were passed through a preconditioned column of Ionac A-580 ion exchange resin which absorbs thorium but allows <sup>228</sup>Ra to go through. This type of chemical separation will be repeated periodically to prevent the buildup of <sup>228</sup>Th in the electromagnetically separated <sup>232</sup>Th.

#### Uranium-235

A program to recover ~500 g of >99.9% <sup>235</sup>U, started in October 1974, is continuing in four singly contained electromagnetic separators. This material is needed for the Interlaboratory LMFBR reaction rate program of the reactor dosimeter center. The charge material (~3000 g of uranium pre-enriched to 97.6% <sup>235</sup>U) is vaporized into the ionization region by reacting CCl<sub>4</sub> with prepared UO<sub>2</sub> positioned in the ion source of the separator, and the separated isotopes are collected in graphite receiver pockets. By the end of the quarter, more than 200 g of <sup>235</sup>U had been monitored. Preliminary mass analyses received to date on 4 of the 11 receiver pockets indicate that the product will be between 99.92 and 99.96% <sup>235</sup>U.

#### Plutonium-244

Near the end of the quarter, a special <sup>244</sup>Pu enrichment project, with the goal of collecting >1 g of ~98% <sup>244</sup>Pu, was completed in one of the doubly contained separators with an estimated collection of 1.16 g of <sup>244</sup>Pu. The initial charge material, enriched to 19.6% in <sup>244</sup>Pu, consisted of 24 g of PuO<sub>2</sub> (21 g Pu). In order to accumulate the stated amount, it was necessary to cycle the charge material through the separator twice, which necessitated a complete charge recovery after the first cycle. The recycled material, consisting of 19 g PuO<sub>2</sub> (16.6 g Pu) and containing 18.3% <sup>244</sup>Pu, became the charge material for the second cycle. A preliminary mass analysis on the first portion collected (0.8 g Pu as unpurified oxide) showed 98.07% <sup>244</sup>Pu.

### CHEMICAL PROCESSING

#### Material in Process

Elements in chemical processing during the quarter are Cd, Ca, Os, Pd, Ru, Se, Te, Sn, and Zr.

#### Material Added to Inventory

Isotopic material released or made available to Sales during the quarter is shown in Table 3, with appropriate separation data. In addition to the nine lots of isotopes released to Sales, 29 loan-return samples were processed and returned to inventory.

Table 3. Operations Status---October-December 1974

| Element<br>and<br>Isotope | Series | Date    |          | Hours  |        | Charge<br>Material                 | Recovery (g) |         | Purity (%) |        | Requirements |            |
|---------------------------|--------|---------|----------|--------|--------|------------------------------------|--------------|---------|------------|--------|--------------|------------|
|                           |        | Start   | Finish   | Innage | Tank   |                                    | Est.         | Actual  | Est.       | Actual | Grams        | Purity (%) |
| <sup>112</sup> Cd         | PQ     | 10/1/73 | 4/5/74   | 21,106 | 24,768 | CdO+CCl <sub>4</sub>               |              | 464.071 |            | 97.05  | 170          | >95        |
| <sup>176</sup> Hf         | PD     | 4/5/71  | 3/30/72  | 10,696 | 15,000 | HfO <sub>2</sub> +CCl <sub>4</sub> |              | 2.061   |            | 72.17  | 92           | >70        |
| <sup>176</sup> Hf         | PD     | "       | "        | "      | "      | "                                  |              | 1.944   |            | 63.88  |              |            |
| <sup>201</sup> Hg         | R&D    |         |          |        |        |                                    |              | 0.746   |            | 96.71  | 11           | ≥90        |
| <sup>201</sup> Hg         | R&D    |         |          |        |        |                                    |              | 0.612   |            | 93.18  |              |            |
| <sup>201</sup> Hg         | R&D    |         |          |        |        |                                    |              | 0.294   |            | 80.58  |              |            |
| <sup>204</sup> Hg         | R&D    |         |          |        |        |                                    |              | 1.067   |            | 94.58  | 46           | ≥90        |
| <sup>189</sup> Os         | PO     | 5/10/73 | 8/29/74  | 3,864  | 5,276  | Os+CCl <sub>4</sub>                |              | 30.937  |            | 94.51  | 25           | ≥87.3      |
| <sup>192</sup> Os         | PO     | "       | "        | "      | "      | "                                  |              | 83.508  |            | 99.06  | 100          | >95        |
| <sup>102</sup> Pd         | PR     | 10/1/73 | 11/14/74 | 3,191  | 4,708  | Pd+ClF <sub>3</sub>                | 1.5          |         | >75        |        | 100          | >80        |
| <sup>104</sup> Pd         | PR     | "       | "        | "      | "      | "                                  | 20           |         | 95         |        | 95           | >90        |
| <sup>105</sup> Pd         | PR     | "       | "        | "      | "      | "                                  | 30           |         | >95        |        | 90           | >90        |
| <sup>106</sup> Pd         | PR     | "       | "        | "      | "      | "                                  | 50           |         | >98        |        | 88           | >90        |
| <sup>108</sup> Pd         | PR     | "       | "        | "      | "      | "                                  | 55           |         | >98        |        | 85           | ≥85        |
| <sup>110</sup> Pd         | PR     | "       | "        | "      | "      | "                                  | 20           |         | >98        |        | 97           | >90        |
| <sup>96</sup> Ru          | PU     | 9/28/74 | 11/14/74 | 586    | 888    | Ru+ClF <sub>3</sub>                | 1.5          |         | >97        |        | 100          | >90        |
| <sup>98</sup> Ru          | PU     | "       | "        | "      | "      | "                                  | 0.75         |         | >85        |        | 100          | >95        |
| <sup>99</sup> Ru          | PU     | "       | "        | "      | "      | "                                  | 4.5          |         | >95        |        | 100          | >90        |
| <sup>100</sup> Ru         | PU     | "       | "        | "      | "      | "                                  | >4           |         | >95        |        | 97           | >95        |
| <sup>101</sup> Ru         | PU     | "       | "        | "      | "      | "                                  | 6            |         | >97        |        | 95           | >95        |

Table 3 cont'd.

| Element<br>and<br>Isotope | Series | Date    |          | Hours  |        | Charge<br>Material  | Recovery (g) |        | Purity (%) |        | Requirements |            |
|---------------------------|--------|---------|----------|--------|--------|---------------------|--------------|--------|------------|--------|--------------|------------|
|                           |        | Start   | Finish   | Innage | Tank   |                     | Est.         | Actual | Est.       | Actual | Grams        | Purity (%) |
| <sup>102</sup> Ru         | PU     | 9/28/74 | 11/14/74 | 586    | 888    | Ru+ClF <sub>3</sub> | 10           |        | ~99        |        | 90           | >95        |
| <sup>104</sup> Ru         | PU     | "       | "        | "      | "      | "                   | 7            |        | ~99        |        | 94           | >95        |
| <sup>40</sup> Ca          | PT     | 4/8/74  | 10/31/74 | 21,719 | 27,840 | Ca metal            | 1900         |        | 99.9       |        | 157          | >99.87     |
| <sup>42</sup> Ca          | PT     | "       | "        | "      | "      | "                   | 9.5          |        | 92         |        | 1.2          | >90        |
| <sup>43</sup> Ca          | PT     | "       | "        | "      | "      | "                   | 2.3          |        | 82         |        | 0            |            |
| <sup>44</sup> Ca          | PT     | "       | "        | "      | "      | "                   | 33           |        | 98         |        | 0            |            |
| <sup>46</sup> Ca          | PT     | "       | "        | "      | "      | "                   | 0.05         |        | ~40        |        | 0.100        | <40        |
| <sup>48</sup> Ca          | PT     | "       | "        | "      | "      | "                   | 2.6          |        | >97        |        | 0.500        | >40        |
|                           |        |         |          |        |        |                     |              |        |            |        | 23           | >94        |

## TARGET FABRICATION AND DEVELOPMENT

Tritium Targets

Requirements for 14-MeV neutron sources with intensities of  $\sim 10^{14}$  n/sec for CTR and fast-reactor experiments necessitate studies involving tritium-containing targets. More than 50 such targets were prepared for use in Livermore deuteron accelerators. Substrates for these targets were tungsten, copper, and "Amzirc" of nominal 15/16-in. dia with a variety of sizes and thicknesses of titanium deposit. These targets will be used for studying neutron-generating characteristics and other physical phenomena involved in the (d,t) reaction and for evaluating the relationship between target design and neutron output.

Plutonium Targets

Several metallic samples of  $^{239}\text{Pu}$  and  $^{240}\text{Pu}$  (15.9-mm dia, with thickness varying from 1.25 mm to 20 mm and with plane-parallel ends) are being prepared for the Neutron Physics Division. Oxides of the two isotopes were sent to Dow Chemical Company, Rocky Flats Facility, for reduction to metal and incorporation of  $\sim 1\%$  aluminum to stabilize the cubic structure so that maximum ductility was achieved. Each isotope metal sample was arc melted and drop cast to obtain a cylindrical bar of slightly larger diameter than the prescribed finished dimension. A small, heavy-duty lathe is being installed in an argon-atmosphere glove box for the purpose of machining the diameter and final lengths of the longer samples from the cast bar. The remaining scrap (turnings plus bar stock) will be remelted and rolled to the thicknesses of the thinnest sample dimensions. This will be the only machine capability of this type, except at LASL, in ERDA laboratories that will permit preparation of high-assay samples for research purposes. It is expected that this capability will permit future fabrication of many actinide elements.

Stable Isotope Targets

The largest order to date for stable-isotope accelerator targets was completed during the quarter, comprising 21 individual targets,  $2.5 \times 2.5 \text{ cm}^2$ , for List-Electronic in Germany; these included isotopes of Ca, Fe, Pb, Mg, Ni, Ti, Cr, and Zn.

Continued effort to develop methods of producing thick targets of isotope metals has permitted fabrication of samples for high-energy-photon experiments, neutron-interaction experiments, and high-energy-charged-particle experiments. Targets of isotopes of many lanthanides, Mg, Mo, Ni, Cu, Fe, Zn, Cd, and alkaline earths, have been prepared, with areas up to  $100 \text{ cm}^2$  and thicknesses of 20 to  $400 \text{ mg/cm}^2$ . In one case, a 1-cm-thick,  $5 \times 5 \text{ cm}^2$  target containing 40 g of  $^{40}\text{Ca}$  was produced for ANL. The metal was produced by reduction--distillation from CaO--La mixtures and arc-melting the distillate into consolidated form. The calcium was finally formed into the square configuration at a pressure of  $8 \text{ tons/cm}^2$ ; six individual anneals and pressings were required.

Targets for Production of  $^{236}\text{Pu}$  and  $^{237}\text{Pu}$  at ORIC

Target development for production of isotopes of plutonium for medical and other uses by high-energy deuteron or alpha bombardment of  $^{235}\text{U}$  involved the bonding of 150-mg/cm<sup>2</sup>- and 650-mg/cm<sup>2</sup>-thick foils of uranium to a nominally 6.3-mm-thick copper substrate by a hot pressure-bonding technique (3000 psi at 700°C); this resulted in a 0.001-mm diffusion layer between the uranium and copper. By this technique, maximum thermal conductivity was achieved so that the 750 watts of imposed ORIC beam energy could be rapidly dissipated to the water-cooling system without target degradation and without separation of the uranium from the substrate. In addition to the copper that bonded the uranium to the substrate, a protective cover of 0.075-mm-thick copper was bonded to the beam side of the uranium by the same method. Targets produced by this method withstood an ORIC 30 MV  $\alpha$ -beam current of 25  $\mu\text{A}$  for 20 hours, which produced about 40  $\mu\text{Ci}$   $^{237}\text{Pu}$ .

## ISOTOPES PREPARATIONS AND SALES

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

Table 4. Stable Isotope Preparations

| Isotope          | No. of Preparations | Isotope            | No. of Preparations |
|------------------|---------------------|--------------------|---------------------|
| Antimony-121     | 1                   | Magnesium-24       | 4                   |
| Normal beryllium | 1                   | -25                | 2                   |
|                  |                     | -26                | 4                   |
| Boron-10         | 1                   | Molybdenum-92      | 1                   |
| Cadmium-114      | 2                   | Nickel-58          | 3                   |
| Calcium-40       | 3                   | -60                | 2                   |
|                  |                     | -62                | 2                   |
| -42              | 1                   | Palladium-105      | 2                   |
| -44              | 3                   |                    |                     |
| -48              | 2                   |                    |                     |
| Chromium-50      | 1                   | Platinum-198       | 1                   |
| -52              | 2                   | Samarium-148       | 2                   |
| -54              | 2                   | -150               | 1                   |
| Dysprosium-160   | 1                   | -152               | 1                   |
|                  |                     | Tin-112            | 1                   |
| -161             | 1                   | -119               | 2                   |
| Erbium-166       | 1                   | -124               | 1                   |
|                  |                     | Titanium deuteride | 12                  |
| Hafnium-176      | 1                   | Titanium-46        | 2                   |
| -179             | 1                   | -48                | 1                   |
| Iron-54          | 2                   | -49                | 1                   |
|                  |                     | -56                | 4                   |
|                  |                     | -57                | 1                   |
|                  |                     | -58                | 5                   |
| Lead-204         | 2                   | -50                | 6                   |
|                  |                     | Ytterbium-168      | 2                   |
|                  |                     | -206               | 3                   |
|                  |                     | Zinc-64            | 2                   |
| -207             | 1                   | -66                | 1                   |
| -208             | 2                   | Zirconium-90       | 1                   |
| Lithium-6        | 101                 | -91                | 2                   |
|                  |                     | -92                | 2                   |
|                  |                     | -94                | 1                   |

Table 5. Radioisotope Preparations

| Isotope         | No. of Preparations | Isotope          | No. of Preparations |
|-----------------|---------------------|------------------|---------------------|
| Berkelium-249   | 1                   | Thorium-232      | 2                   |
| Californium-252 | 1                   | Tritium-titanium | 156                 |
| Carbon-14       | 2                   | Normal uranium   | 1                   |
| Curium-244      | 12                  | Uranium-235      | 2                   |
| Plutonium-239   | 153                 |                  |                     |

Table 6. Isotopes Sales and Services

|  | Revenue        | No. of Shipments |
|--|----------------|------------------|
| EM Isotopes Sales  | \$305,456      | 693              |
| Non-EM Isotopes Sales                                      | 12,470         | 99               |
| Heavy Elements and<br>Other Special Materials              | 73,748         | 104              |
| Services   |                |                  |
| Stable Isotope Target Preparations                         | \$ 44,241      |                  |
| Radioisotope Target Preparations                           | 14,635         |                  |
| Heavy Elements and Other Special<br>Materials Preparations | 34,385         |                  |
| Special Services   | 7,825          |                  |
| Reprocessing Returned Loans                                | 12,463         |                  |
| Work for Others  | <u>17,356</u>  |                  |
| Total Services   | <u>130,905</u> |                  |
| Total  | \$522,579      | 896              |

## ADMINISTRATIVE

Visitors to the Isotopes Development Center are shown below.

| Visitor       | Affiliation          | Subject Discussed               |
|---------------|----------------------|---------------------------------|
| Mel Coops     | LLL<br>Livermore, CA | Fission chamber<br>preparations |
| Paul DeBievre | BCMN<br>Mol, Belgium | Target preparation              |

## PUBLICATIONS

G. Vourvopoulos, M. B. Greenfield, J. B. Ball, S. Raman, and W. K. Dagenhart, Two-Step Mechanism in the  $^{22}\text{Ne}(\rho, t)$  Reaction, *Physics Letters*: 52B(2): 187-8 (Sept. 30, 1974).

J. H. Gillette, *Isotope Program (NT) Progress Report for Quarter Ending Sept. 30, 1974*, ORNL-TM-4745 (Dec. 1974).



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