MANUAL OF ISOTOPE PRODUCTION PROCESSES
IN USE AT BROOKHAVEN NATIONAL LABORATORY

Louis G. Stang, Jr., Coordinator

August 1964

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MANUAL OF ISOTOPE PRODUCTION PROCESSES
IN USE AT BROOKHAVEN NATIONAL LABORATORY

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August 1964
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FOREWORD

One purpose of this report is to collect together the various procedures used at Brookhaven National Laboratory (Hot Laboratory Division, Nuclear Engineering Department) for producing those isotopes which require chemical processing after they are formed by irradiation. Isotopes which are produced by simple irradiations and require no further processing are supplied by a different group (Isotopes and Special Materials Group, Reactor Division) and are not treated here. A second purpose, in response to several requests, is to describe the actual techniques in more detail than is usual in most publications. The essential information contained here has already been made available in the open literature, as shown by the references listed following the Introduction, which also provide background theory and discussion not appropriate for inclusion in this report.

Each procedure begins with a listing of certain facts about the isotope or procedure. In most cases literature values are used for half-life, cross section, fission yield, and radiations. Note that the values listed are not necessarily the "best" values, nor are they necessarily correct to the number of significant figures given. Rather they are the values which we, ourselves, might use in our work and are listed here to provide useful, relevant information. Note also that some radiations other than the ones listed exist.

The values shown under target dimensions and/or weight, neutron flux, beam energy, irradiation time, and integrated beam current are typical values which we actually use, and those shown under specific activity, product composition, radiochemical purity, and yields (other than fission yield) are typical results obtained under these conditions.

This report is the product of the work of several individuals in the Hot Laboratory Division. Most of the work on developing "new" isotopes is carried out by the Isotopes Research Group, headed by W. D. Tucker until 1961 and since then by Manny Hillman. Most of the work of putting these procedures into practice and of actually producing the isotopes (for which Brookhaven is in many cases the world's sole supplier) is carried out by the Operations Group headed by Powell Richards. The line of demarcation is not always sharp; occasionally the Research Group may prepare an infrequently needed isotope, and more often the Operations Group may develop improvements either in production methods or even in ways of using particular isotopes. Names of people who are or have been actively engaged in this work can be inferred from the list of references. Without their work and their help this report would have been impossible. The capable assistance of Grace Searles in preparing the final copy is also gratefully acknowledged.

L. G. Stang, Jr.
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INTRODUCTION

No attempt is made in this report to define terms or provide explanations of phenomena and operations in general chemistry, radiochemistry, and handling of radioactive material via remotely operated equipment, since the reader is assumed to be familiar with these areas. However, instructions are sufficiently detailed to permit others to reproduce our procedures and our results without further development work. A brief description of our facilities and specialized equipment is given to orient the reader and provide a better understanding of the way these procedures are carried out.

Except for $^{48}$Sc all the isotopes described in this report are produced by irradiation either in a thermal neutron reactor or in a cyclotron; a Cockcroft-Walton generator is used to make the fast neutrons needed for producing $^{48}$Sc. The reactor is a graphite-moderated, air-cooled reactor providing a maximum thermal neutron flux of $\sim 1.3 \times 10^{13}$ n cm$^{-2}$ sec$^{-1}$. The cyclotron is a fixed-frequency machine with 62-in.-diameter pole faces capable of delivering alpha particles, deuterons, and protons of 42, 21, and 10.5 MeV, respectively, in external beam currents of 30 to 50 $\mu$A or internal beam currents of $\sim 300$ $\mu$A. (In practice many targets present cooling problems which require limitation of beams to $\sim 50$ $\mu$A.)

Irradiated targets which require heavy shielding are brought from the reactor in a transfer shield. Figure 1 shows such a shield positioned at the reactor with the central hole horizontal ready for loading. When the loaded shield is rotated 90° counterclockwise so that the central hole is vertical, and the transfer drawer is opened, the irradiated target falls out.

Originally this operation was carried out over an opening in the roof of a hot cell (Figures 2 and 3) and the sample dropped directly into a dissolver vessel (Figure 4) positioned under the opening; in this case both the sample and the aluminum can surrounding it were dissolved. Recently a lead brick cave accommodating simple ball-swivel tongs, a remotely operated vise, and a remotely operated pipe cutter was built over the roof opening of the cell; when the irradiated target is dropped into the cave, the sample container can be removed mechanically and the sample dropped by itself into the dissolver vessel in the cell below. This vessel has a large opening in its top plate that is closed by a pneumatically operated cover.

Typical processing equipment, as Figures 3 and 4 show, consists of a series of glass vessels interconnected with plastic tubing closed by miniature

valves that are pneumatically controlled by other valves on the control panel. Liquids are transferred by pressure and vacuum applied at the appropriate places rather than by pouring. The glass vessels are made of suitable sections of heavy-wall Pyrex pipe fittings. On particularly useful type of vessel* is shown in Figure 5; air pressure applied under a porous glass disc located near the bottom maintains liquid above the disc, preventing the liquid from seeping through; a change from air pressure to vacuum under the disc causes the solution to filter through to the next vessel. Infrared lamps shining on the outsides of the vessels are used for heating. Reagents are added by pouring into a vessel on the control panel, connecting this vessel to the desired vessel inside, and forcing the reagent over into the reaction vessel by applying air pressure to the reagent addition vessel. This type of equipment and the cells which house it have been described elsewhere** in more detail.

This equipment is particularly useful for separating a few curies of individual fission products from several hundred curies of irradiated U\textsuperscript{235}. Where less activity is to be handled, specifically one curie or less, semiremotely controlled manipulations in a shielded bench of the type shown in Figures 6 and 7 are convenient. Here equipment and active material can be readily introduced through the ends of the shielding or through a hole in the center of the heavy steel table which supports the lead bricks and itself acts as a shield for the operator's legs and feet. A simple rectilinear manipulator (BNL Model #3 shown in the figures) and ball-swivel tongs used together provide satisfactory, if somewhat restricted, manipulation for physical operations and chemical processing. A useful device employed in this type of shielded bench is the cutoff wheel shown in Figure 8.

Figure 9 shows how cyclotron targets are disassembled. It also shows the edge of a target holder with an aluminum ring (on top) which is screwed against the water-cooled holder and clamps aluminum foil over the actual target, which lies in a recess in the holder.


Figure 1. Slug transfer shield.
Figure 2. Hot cell for chemical processing.
Figure 3. Remotely operated chemical processing equipment suspended in front of hot cell ready to be placed inside.
Figure 4. Remotely operated chemical processing equipment (side view) shown removed from hot cell. Removable shielding block fits into stepped opening in shielding doors. Control panel (not shown) would be at left, fastened to removable block. Stainless steel diaphragm just fits cell opening and seals cell when gasket is inflated, preventing accidental leakage of radioactive material to operating side.
Figure 5. Reaction vessel.
Figure 6. Semi-remote manipulation of fractional curie amounts of activity.
Figure 7. Semi-remotely operated equipment behind shielding shown in Figure 6. Turntable positions samples over hot plate-magnetic mixer, under reagent addition line, under pipet, etc. Rectilinear manipulator and ball-swivel tongs use identical interchangeable jaws.
Figure 8. Remotely operated cutoff wheel for glass or quartz. Crank operated flexible shafts position wheel vertically and laterally. Vertical chuck is driven slowly by a separate motor.
Figure 9. Remotely operated target disassembler. Rotating top shaft rotates screwdriver bit. Rotating second shaft tightens or loosens vise; pushing or pulling second shaft moves entire vise to left or right. Rotating third shaft moves vise forward and back. Rotating bottom shaft raises and lowers vise.
REFERENCES


Aluminum-28

Isotope: \( \text{Al}^{28} \)

Shipping Form: Generator

Parent: \( \text{Mg}^{28} \)

Half-Lives: \( \text{Al}^{28}, 2.24 \text{ m}; \text{Mg}^{28}, 20.9 \text{ h} \)

Production Method: See Magnesium-28

Generator Yield: \( \sim 30\% \) if carrier is added; \( <30\% \) without carrier

Specific Activity: From "carrier-free" \( \text{Al}^{28} \) (i.e. no Al carrier added) to any lower value, as desired.

Product Composition: \( \text{NaAl}^{28}\text{O}_2 \) in \( 1 \text{ N NaOH} \)

Radiochemical Purity: \( >99.9\% \)

Method of Analysis: Gross decay and gamma spectroscopy

Radiations (MeV):

\[
\begin{array}{ll}
\text{Beta (Al}^{28}\text{)} & \text{Gamma (Al}^{28}\text{)} \\
2.87 & 1.78 \\
\text{Beta (Mg}^{28}\text{)} & \text{Gamma (Mg}^{28}\text{)} \\
0.42 \ (100\%) & 0.032 \ (96\%) \\
& 0.40 \ (31\%) \\
& 0.95 \ (29\%) \\
& 1.35 \ (70\%) \\
\end{array}
\]

Special Equipment: Generator (see Figure 10)

---

A. Preparation of Generator

1. Over Dowex-50W (\( \text{H}^+ \) form) pass 1 - 2 M \( \text{NaCl} \) until effluent is at same pH as \( \text{NaCl} \). This converts the resin to the \( \text{Na}^+ \) form.

2. Wash resin with \( \text{H}_2\text{O} \) until all \( \text{Cl}^- \) is removed. Use \( \text{Ag}^+ \) to test for presence of \( \text{Cl}^- \).
3. Transfer resin to a specially
made funnel-top glass vessel
shown in Figure 10,
using enough resin to form a
column 0.6 cm high.

![Figure 10. Aluminum-28 generator.](image)

B. Repurification of Mg\(^{28}\)

1. Start with Mg\(^{28}\)Cl solution.

2. Add ~5 mg each of Cu\(^{++}\) and
Cd\(^{++}\) carriers.

3. Dilute to 15 ml and adjust pH
to 3 - 4 with 0.1 N NaOH
solution.

4. Saturate with H\(_2\)S gas and
digest on a hot plate.

5. Filter through paper placed
on glass frit on filter chimney.

6. To filtrate add a few drops of
Brom Thymol Blue indicator.

This is supplied by the Hot
Laboratory Division, Brookhaven
National Laboratory. (See
separate procedure for producing
Mg\(^{28}\).)

Minute traces of Cu\(^{64}\) and Cd\(^{115}\)
which will not be detected in the
presence of Mg\(^{28}\) will show up in
the milked Al\(^{28}\) and must be
removed.

Indicator paper is satisfactory.
Too much NaOH will precipitate
Mg(OH)\(_2\).  

Several minutes are required to
form coarse particles of Cu\(^{64}\)S
and Cd\(^{115}\)S.

Use Whatman \#42 paper and fine frit.
Save filtrate; discard precipitate.

Use 0.1% solution.
7. Adjust pH to just acid using 0.1 N NaOH and 0.1 N HCl. Add NaOH dropwise with stirring until blue-green color forms; then add one or two drops HCl until yellow color just forms. Avoid precipitating Mg(OH)₂.

8. Heat for several minutes. Cool. Drive off H₂S.

C. Loading the Generator

1. Add solution from step #8 (above) to 30 ml beaker containing ~0.5 cc Dowex-50W resin in Na⁺ form. Prepare resin as in steps #1 and #2, section A (above).

2. Stir until ≥95% of the Mg²⁺ is adsorbed. This will require several minutes. Check for degree of adsorption by measuring radiation level of resin and supernate separately.

3. Decant the supernate and save. Pour the resin into the generator and cover with glass wool, powdered glass, glass wool, and a polyethylene retaining ring, in that order. See Figure 10 for amount of powdered glass to use. Because of capillary action the powdered glass acts as a shut-off valve and prevents the resin from draining dry if the stopcock is inadvertently left open. (For proper operation it is important to exclude any gas phase from the resin.)

4. Pour the supernate from step #3 into the generator at a slow rate (~1 ml/min). This is to adsorb any Mg²⁺ left in the supernate. (The batchwise adsorption deposits the Mg²⁺ on a relatively large surface area of resin and produces higher subsequent milking yields than would be obtained if all of the Mg²⁺ were adsorbed by passing it through a column.)

5. Pass 200 ml 1 N NaOH through the generator at a rapid rate (5 - 10 ml/min). This washes out any remaining impurities.

D. Milking the Generator

1. Elute Al³⁺ with 2 - 3 ml 1 N NaOH. Alternatively, the NaOH may contain carrier Al³⁺ as desired (e.g. 0.5 mg/ml). Milking requires 10 - 30 seconds. If carrier Al³⁺ is used, yields after the first few milkings will be ~30%. Yields are somewhat lower on the first few milkings or without carrier.
Argon-38

Isotope: \( \text{Ar}^{38} \)

Shipping Form: Gas sealed in glass ampoule

Half-Life: \( \infty \) (stable)

Production Method: \( \text{Cl}^{37}(n,\gamma)\text{Cl}^{38} \xrightarrow{\beta^-} \text{Ar}^{38} \)

Production Cross Section: 0.56 b

Target: NaCl crystals, optical grade, grown under He atmosphere, and wrapped in Al foil

Target Weight: 300 g

Neutron Flux: \( 10^{13} \text{ n sec}^{-1} \text{ cm}^{-2} \)

Irradiation Time: 56 d (excluding any "down time")

Reactor Yield:

Chemical Yield: 0.45 cc (STP) = overall yield

Product Composition: Ar gas. Typical analysis:
- Mass 38, 99.5%
- Mass 40, 0.46%
- Mass 36, 0.035%
- Mass 39, 0.005%

Radiochemical Purity: See above for \( \text{Ar}^{39} \). No radiation detectable outside glass ampoule. Classed as non-radioactive by Interstate Commerce Commission regulations.

Method of Analysis: Mass spectrograph

Special Equipment: High vacuum (~10^{-7} torr) glass gas train incorporating furnace and Vycor vessel for melting NaCl, heated Ag and Ti getters, and the usual pumps, traps, McLeod gauges, and mercury U-tube valves. Bake-out oven capable of surrounding entire train.
A. Post-Irradiation Processing

1. After waiting for Na$^{24}$ to decay, unwrap the Al foil from the irradiated NaCl and transfer $\sim$100 g of the crystals through the inlet port into a Vycor liner in the melting vessel attached to the gas train. Reseal the inlet.

2. Evacuate the system for 24 hours.

3. Heat the Ag getter to $\sim$230°C and the Ti getter to $\sim$1000°C, and bake the entire system at $\sim$370°C for 5 - 8 hours. During the last hour play an air-gas flame over the entire system occasionally.

4. Turn off the Ti and Ag getter furnaces. Then turn off the bake oven.

5. Measure the pressure of the system at room temperature.

6. Close the valves which isolate the sample collection and pump sections from the rest of the system.

7. Heat the Ti getter to 880° - 950°C, the Ag getter to 170° - 180°C, and the NaCl to 800° - 850°C.

8. After $\sim$1 hour refrigerate the charcoal trap with liquid $N_2$ for $\sim$30 minutes.

9. Close the valve between the charcoal trap and the Ag getter. Turn off the furnaces on the Ag getter and the NaCl.

10. Remove the liquid $N_2$ from the charcoal trap, allowing it to return to room temperature while continuing to heat the Ti getter.

Caution: A considerable amount of $S^{36}$ from Cl$^{36}$ (n,p)$S^{35}$ will still be present. 100 g is a convenient amount to process at one time. Freezing NaCl may crack a Vycor container: hence, the inner liner to protect the outer vessel.

This readies the getters by removing whatever will come off at high temperatures; it also removes adsorbed impurities from the rest of the system. The Ag getter is located between the NaCl melting vessel and the main train; it removes $S^{36}$. The Ti getter is on a side arm off the main train.

This sequence prevents readsorbing in the rest of the system any condensibles remaining in the getters.

Pressure should be $\sim$10$^{-7}$ torr.

At $\sim$650°C NaCl starts subliming into the cooler part of the system.

This trap is located in the side arm between the Ti getter and the main line. This operation adsorbs Ar.
11. After \(\sim\)90 minutes, allow the Ti getter to cool to \(\leq 100^\circ\text{C}\).

12. Refrigerate the charcoal trap with liquid \(\text{N}_2\) for \(\sim\)30 minutes more.

13. Open the valve connecting the pumps to the Ti getter-charcoal trap side arm and wait until the pressure becomes constant.

14. Close this valve (step #12) and replace the liquid \(\text{N}_2\) around the charcoal trap with a dry ice-solvent mixture.

15. Open the valve to the sample collection section and refrigerate with liquid \(\text{N}_2\) the charcoal trap in that section.

16. When the pressure has become constant close this valve (step #14).

17. Remove liquid \(\text{N}_2\) from sample collection charcoal trap, allowing it to return to room temperature.

18. Measure pressure and temperature and from known volume of system calculate amount of Ar present.

19. Seal off glass ampoules.

This removes residual gaseous impurities from the system.

Ar desorbs at dry ice temperature but higher boiling impurities do not.

This transfers the Ar to the sample collection section; the volume of this section should be known in advance.

\(\sim\)50 ml is a convenient size for each ampoule. Ampoules have the usual break-off seal permitting the ampoule to be sealed to a new system and the contents to be admitted thereto.
Copper-67

Isotope: Cu$^{67}$
Half-Life: 61.6 h
Production Method: Ni$^{64}$(α,p)Cu$^{67}$ (cyclotron)
Target: Ni$^{64}$, enriched to 95%
Target Dimensions: 0.5 x 0.25 x 0.005 inches
Beam Energy: ~40 MeV
Integrated Beam Current: 725 μAh; (14.5 h at 50 μA)
Cyclotron Yield: 1.7 mC
Chemical Yield: >95%
Specific Activity: >1.7 mC/mg
Product Composition: Cu$^{67}$(NO$_3$)$_2$ in 6 M HNO$_3$
Radiochemical Purity: >99% as of 3 days cooling
Method of Analysis: Gamma spectroscopy and gross decay
Radiations (MeV):

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<td>0.092</td>
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<td>0.484</td>
<td>0.182</td>
<td></td>
</tr>
<tr>
<td>0.577</td>
<td>0.296</td>
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Special Equipment: Electrolysis apparatus.

A. Target Preparation and Irradiation

1. Silver solder gold foil onto a copper target blank.

2. Electroplate enriched Ni$^{64}$ from ammoniacal solution onto gold foil, with stirring.

Blank must be capable of being water-cooled during irradiation. Good contact between gold and copper is required for heat transfer.

(NO$_3$)$^-$ and Cl$^-$ must be absent. Anode may be graphite. Stirring prevents local depletion of Ni$^+$ at cathode.
3. Bombard target in the internal beam of the cyclotron.

Caution: >50 μA will melt gold foil.

B. Isolation of Cu^{67}

1. Unsolder gold foil.

2. Place foil in 50 ml beaker, nickel side down on a few drops of hot \( \text{HNO}_3 \).

3. After Ni has dissolved, rinse foil with \( \text{H}_2\text{O} \), add 3 drops conc. \( \text{H}_2\text{SO}_4 \), and transfer quantitatively to a special electrolysis vessel clamped in a warm (60°C.) water bath.

4. Insert platinum wire electrodes.

5. Add \( \sim 0.5 \text{ mg} \) each of Co^{++}, In^{+++}, Cd^{++}, and Cu^{++}.

6. Apply 2.4 - 2.6 V DC to electrodes for \( \sim 45 \text{ m} \).

7. Without reducing applied voltage remove cathode and promptly rinse with \( \text{H}_2\text{O} \).

8. Dissolve product by running 1 - 2 drops 6 M \( \text{HNO}_3 \) down the wire.

C. Reclamation of Ni^{64}

1. Make Ni solution basic with an excess of conc. \( \text{NH}_3\text{OH} \).

2. Filter and discard any precipitate.

3. To filtrate add solution of 0.01 N dimethylglyoxime.

4. Digest near boiling.

This removes ions which interfere with subsequent preparation of electrolysis solution.

This precipitates nickel dimethylglyoxime.

This permits particle growth and easier subsequent filtration.
5. Cool, filter, and wash, discarding filtrate.

6. Place the filter paper and the precipitate in a beaker and add 40 ml conc. HNO₃ and 15 ml conc. HClO₄.

7. Evaporate to dryness.

8. Dissolve residue in a minimum of 9 N H₂SO₄ and neutralize with concentrated NH₄OH.

Filter through paper.

This will completely oxidize and dissolve both precipitate and paper, volatilizing organic components.

Solution is ready for electroplating a new target.
Fluorine-18

Isotope: \(^{18}\text{F}\)

Half-Life: 110 m

Production Method: \(\text{Li}^6(n,t)\text{He}^4; \text{O}^{16}(t,n)^{18}\text{F}\) (reactor)

Cross Section: 950 b for \(\text{Li}^6(n,t)\text{He}^4\)

Target: \(\text{Li}_2\text{CO}_3\) (unenriched)

Target Weight: 10 g

Neutron Flux: \(1.2 \times 10^{13}\) n sec\(^{-1}\) cm\(^{-2}\)

Irradiation Time: \(~6\) h

Reactor Yield: \(~40\) mC

Chemical Yield: 70 - 80%

Specific Activity: "Carrier-free" \(^{18}\text{F}\) (i.e., no F carrier added)

Product Composition: \(\text{NaF}^{18} + \text{trace of NaAlO}_2\) in 0.1 M NaOH

Radiochemical Purity: >99.99% \(^{18}\text{F}\); trace of \(^{18}\text{O}\)

Method of Analysis: Well-type scintillation counter standardized against 4\(\pi\) B counter

Radiations (MeV): Positron \(0.65\) (100%)  Gamma \(0.51\) (annihilation gammas)

Special Equipment: Glove box or fume hood suitable for handling \(^{18}\text{F}\) contamination. Isotope can opening tools and crimping press for closing. 1/4-inch shielding locally.
A. Target Preparation

1. Insert an aluminum tube coaxially inside a standard aluminum isotope can and pack the annular space with $\sim 10$ g $\text{Li}_2\text{CO}_3$.

Annular space should be $\sim 1/8$ inch thick. Because of the high $\sigma$ of $\text{Li}^6$, if the annular layer were appreciably thicker than this, the internal neutron flux would be so depressed that the material inside would contribute little or no additional product and would only add undesirable bulk to the process.

2. Crimp top of can.

Top should have small hole covered with Scotch tape to prevent pressure build-up.

B. Post-Irradiation Processing

1. Remove top from isotope can and transfer $\text{Li}_2\text{CO}_3$ powder to Erlenmeyer flask containing $\sim 20$ ml $\text{H}_2\text{O}$ and magnetic stirring bar.

Use wide neck funnel and rinse with $\text{H}_2\text{O}$.

2. Add conc. HCl slowly with stirring to dissolve $\text{Li}_2\text{CO}_3$.

Avoid rapid $\text{CO}_2$ evolution.

3. Adjust pH to acid side of Brom Thymol Blue indicator with NaOH solution.

Column preparation: Slurry 10 g Woelm alumina (neutral grade) with 50 ml 0.5 M HCl, decanting and discarding fines. Wash with two 50-ml portions of $\text{H}_2\text{O}$, discarding fines. To a 1-cm diameter glass tube to which is sealed a coarse frit, transfer enough alumina to stand 3 cm above the frit. Wash a few times with $\text{H}_2\text{O}$. A pad of glass wool on top of the alumina will prevent it from plugging.

4. Pass solution through alumina column, using vacuum to attain 1 - 2 ml/min.

Use vacuum if necessary.

5. Wash column with 30 ml $\text{H}_2\text{O}$ containing 1 drop conc. HCl.

Collect portions in large test tubes inside side-arm flask on which slight vacuum is pulled.

6. Elute $\text{F}^8$ with 40 ml 0.1 N NaOH containing Brom Thymol Blue indicator. Collect in 3 portions:
7. Assay portion #2 and transfer to shipping bottle.

Note: Product is an aluminum fluoride complex, which may be converted to NaF by distillation from conc. H₂SO₄, collecting distillate in dilute NaOH.


**Galium-68**

Isotope: \( \text{Ga}^{68} \)

Shipping Form: Generator

Parent: \( \text{Ge}^{68} \)

Half-Lives: \( \text{Ga}^{68} \), 68 m; \( \text{Ge}^{68} \), 275 d

Production Method: \( \text{Ga}^{69}(p,2n)\text{Ge}^{68} \) (ORNL cyclotron; see Isotope Sales Department, ORNL, for details)

Generator Yield: \( \sim 70\% \)

Specific Activity: Carrier-free

Product Composition: \( \text{Ga}^{68} \)-EDTA complex in 0.005 M EDTA; or alternatively as \( \text{Ga}^{68}\text{Cl}_3 \) in ether; or alternatively as \( (\text{Ga}^{68})_2(\text{SO}_4)_3 \) "weightless" solid.

Radiochemical Purity: 99.999\% \( \text{Ga}^{68} \); 0.001\% \( \text{Ge}^{68} \)

Method of Analysis: Gross decay

Radiations (MeV):

- **Positron (Ga\(^{68}\))**
  - 0.82 (1.5\%)
  - 1.894 (86\%)

- **Gamma (Ga\(^{68}\))**
  - 0.51 (annihilation gammas)
  - 1.08 (~4\%)

- **Particle (Ge\(^{68}\))**
  - none
  - \( \text{Ga}^{68} \) only characteristic \( \text{Ga}^{68} \) X rays; (100\% electron capture)

---

**A. Preparation of Generator**

1. Wash 10 g alumina with \( \text{H}_2\text{O} \) and slurry into a glass cylinder (~25 mm diameter) having a sealed-in glass frit. This removes fines. Use Fisher or "Woelm" chromatographic alumina. Use coarse porosity glass frit.

2. Place a retaining plug of glass wool or glass cloth over the alumina. This insures mechanical stability of the alumina during subsequent handling.
3. Pass ~50 ml 0.005 M neutralized EDTA through the alumina. EDTA is the common designation of ethylenediaminetetracetic acid. Before use in this procedure its pH should be adjusted to 7 with NaOH.

B. Loading the Generator

1. Start with a solution of Ge$^{68}$ as the chloride. This is available commercially.

2. Add sufficient EDTA to give a final concentration of 0.005 M and a final volume of ~500 ml. Solution as received commercially is strongly acid.

3. Adjust pH to 7.

4. Pour onto the alumina in the generator.

5. Pass 100 ml EDTA solution (pH 7) through the alumina.

C. Milking the Generator

1. Elute Ge$^{68}$ with 25 ml neutralized 0.005 M EDTA. Collection rate should be 25 ml/3 - 5 min.

Note: If EDTA in the product is undesirable it may be removed by (1) extracting Ge$^{68}$ from 6 M HCl into ether and back extracting into $H_2O$, or (2) adding 1 - 2 drops conc. $H_2SO_4$ and 1 - 2 ml $HNO_3$ and evaporating solution to dryness.
Iodine-124

Isotope: $^{124}$I

Half-Life: 4.2 d

Production Method: Sb$^{121}$($\alpha$,n)$^{124}$ (cyclotron)

Target: Sb (unenriched, pressed powder)

Target Dimensions: 5.5 x 0.75 x 0.005 inches

Beam Energy: ~40 MeV

Integrated Beam Current: 494 µAh; (13 h at 38 µA)

Cyclotron Yield: 9.5 mC $^{124}$I after 48 h cooling

Chemical Yield: ~80%

Specific Activity: ~2 C $^{124}$I/g I$^-$ as of 48 h cooling

Product Composition: $^{124}$I in NaHSO$_3$ solution or in Na$_2$SO$_3$ solution

Radiochemical Purity: >89% $^{124}$I, <10% $^{123}$I, <1% $^{125}$I as of 48 h cooling

Method of Analysis: Gamma spectroscopy and gross decay

Radiations (MeV):

<table>
<thead>
<tr>
<th>Positron</th>
<th>Gamma</th>
</tr>
</thead>
<tbody>
<tr>
<td>~0.90 (0.2%)</td>
<td>0.603 to</td>
</tr>
<tr>
<td>1.55 (14%)</td>
<td>2.15 (11%)</td>
</tr>
<tr>
<td>2.15 (11%)</td>
<td>2.745</td>
</tr>
</tbody>
</table>

>19 γ's

Special Equipment: Press capable of exerting force of 60 - 70 tons. Suitable die. Semi-remotely operated distillation equipment and target handling equipment.

A. Target Preparation

1. Weigh out 7 g powdered Sb into a suitable die.

Die area: 5.5 x 0.75 inches.

2. Press.

A force of 60 - 70 tons is required for proper compaction.
3. Place in special water-cooled holder.

4. Cover target with aluminum foil; bolt on retaining frame.

Holder is a flat metal plate with a recess to fit target and a bolted-on retaining frame.

Use 2.5-mil foil. Thinner will melt; thicker will degrade α energy too much.

B. Post-Irradiation Processing

1. Score and cut the aluminum foil around the inside of the retaining frame.

2. Remove and discard foil and dump target into still pot containing chromic acid-sulfuric acid-iodide carrier mixture.

3. After target dissolution cool solution and add 100 ml H₂O and 10 ml 30% H₃PO₃.

4. Heat and distill into H₂SO₃ solution, adding 1 - 2 drops 30% H₂O₂ occasionally.

5. To distillate add 1 g oxalic acid and 18 - 20 ml conc. H₂SO₄ and redistill into H₂SO₃ solution.

6. If desired, the product may be further concentrated by making basic and evaporating down.

Use a manipulator-held scribing point.

Use tweezers or suitable manipulator.

Mixture preparation: Dissolve 13 g CrO₃ in 70 ml H₂O; add 280 ml conc. H₂SO₄ and 5 μg I⁻ carrier.

This reduces the iodates to a distillable form.

H₂O₂ releases O₂ gas which acts as a carrier. H₂SO₃ traps radiiodines in non-volatile form. Stop distillation when activity ceases to distill.

This removes any Sb carried over during the first distillation. No H₂O₂ is needed here because of small volumes.

Use NaOH solution to make basic.

This product will bind to protein.

**Note:** If product is not going to be bound to protein it can be distilled directly into NaOH instead of into H₂SO₃.
Iodine-132

Isotope: $^{1^{32}}I$

Shipping Form: Generator

Parent: $Te^{132}$

Half-Lives: $^{1^{32}}I$, 2.3 h; $Te^{132}$, 77 h

Production Method: See Tellurium-132

Generator Yield: $\sim 70\%$

Specific Activity: Carrier-free

Product Composition: Na$^{1^{32}}I$ in 0.01 M $NH_4OH$; traces of stable Al and Na

Radiochemical Purity: <10$^{-3}\%$ $Te^{1^{32}}I$; <0.01% - 0.3% $I^{131}$, depending on history of generator (see note below)

Method of Analysis: Well-type scintillation counter standardized against $4\gamma \beta$ counter

Radiations (MeV):

<table>
<thead>
<tr>
<th>Beta ($^{1^{32}}I$)</th>
<th>Gamma ($^{1^{32}}I$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.80 (21%)</td>
<td>0.528</td>
</tr>
<tr>
<td>1.04 (15%)</td>
<td>0.670</td>
</tr>
<tr>
<td>1.22 (12%)</td>
<td>0.777</td>
</tr>
<tr>
<td>1.49 (12%)</td>
<td>0.96</td>
</tr>
<tr>
<td>1.61 (21%)</td>
<td>and &gt;23 other $\gamma$'s from 0.240 to 2.9</td>
</tr>
<tr>
<td>2.14 (18%)</td>
<td></td>
</tr>
<tr>
<td>2.92 (0.03%)</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Beta ($Te^{1^{32}}$)</th>
<th>Gamma ($Te^{1^{32}}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22</td>
<td>0.052</td>
</tr>
<tr>
<td></td>
<td>0.233</td>
</tr>
</tbody>
</table>

Special Equipment: Remotely operated dispensing equipment.

A. Preparation of Generator

1. Load a glass Buchner funnel with
   10 ml pre-washed Fisher chromatographic alumina.

   Pre-wash alumina with $H_2O$, decanting fines. Use a 25 mm dia. x 85 mm high funnel.
2. Cover with Feon cloth, held down by a polyethylene retainer ring. "Feon" is a non-absorbing acrylic fiber filter cloth.

3. Wash with H\textsubscript{2}O until effluent is clear.

4. Pass 50 ml 1 M HNO\textsubscript{3} through the alumina.

B. Loading of Generator

1. Into a few ml H\textsubscript{2}O pipet the required amount of Te\textsuperscript{132} product solution and make up volume to 25 ml with H\textsubscript{2}O. Use solution from step #25 of Te\textsuperscript{132} process.

2. Mix well. Remotely operated magnetic stirrer is convenient.

3. Drip into generator (see above) at \(<1\) drop/sec. This neutralizes any residual free acid left on the alumina.

4. Pass 30 ml H\textsubscript{2}O through generator.

5. Pass 10 ml 1 M NH\textsubscript{4}OH through generator.

6. Pass 500 ml 0.01 M NH\textsubscript{4}OH through generator.

Note: Generator is milked (i.e. Te\textsuperscript{132} is eluted from alumina) with 10 - 20 ml 0.01 M NH\textsubscript{4}OH. Washing the resin with 0.01 M NH\textsubscript{4}OH prior to a given milking decreases the Te\textsuperscript{131} contamination in that milking. This decrease is a direct function of the amount of wash solution used, but volumes over 300 ml of wash solution produce so little further decrease in contamination that 300 ml can be considered the practical maximum volume desirable. The Te\textsuperscript{131} contamination also decreases with increasing age of generator and with increasing "growth time" (defined as the time between a given milking and the previous milking or wash). Thus, for minimum Te\textsuperscript{131} contamination one would choose an old generator and wash it with 300 ml 0.01 M NH\textsubscript{4}OH at whatever time prior to milking would allow only the desired amount of Te\textsuperscript{132} to grow in (i.e. at that time which provides the minimum growth time for the Te\textsuperscript{132} desired).
Iodine-133

Isotope: $^{133}\text{I}$

Half-Life: $^{133}\text{I}, 21 \text{ h}; \text{Xe}^{133} \text{ (daughter), 5.3 d}$

Production Method: $^{235}\text{U}(n,f) \text{ (reactor)}$

Fission Cross Section: 582 b

Target: $\text{U}_3\text{O}_9 \text{ (unenriched)}$

Target Weight: 2 g

Neutron Flux: $\sim 1.0 \times 10^{13} \text{ n sec}^{-1} \text{ cm}^{-2}$

Irradiation Time: 20 h

Fission Yield: $\sim 6.9\%$

Reactor Yield: This process produces $\sim 100 \text{ mCi}^{133}\text{I}$.

Chemical Yield: "Carrier-free" $^{133}\text{I}$ (i.e., no I carrier added)

Specific Activity: $^{133}\text{I}_{\text{specific}} = 1.0 \times 10^{13} \text{ disintegration sec}^{-1} \text{ cm}^{-2}$

Product Composition: $\text{NaI}^+ + \text{NaI}^+\text{O}_3$ in dilute NaOH

Radiochemical Purity: $^{131}\text{I},^{135}\text{I}$ also present. At 30 h after removal from reactor the composition is $\sim 78\%$ $^{133}\text{I}$, 8\% $^{131}\text{I}$, and 14\% $^{135}\text{I}$. During the next 40 h $^{133}\text{I}$ constitutes $\geq 75\%$ of the total radioiodine activity. At 105 h the $^{133}\text{I}$ activity equals the $^{131}\text{I}$ activity.

Method of Analysis: 4$n^\beta$ count to determine total activity at reference time; gross decay curve on previous batches to determine relative proportions of various radioiodines.

Radiations (MeV):

<table>
<thead>
<tr>
<th>Beta ($^{133}\text{I}$)</th>
<th>Gamma ($^{133}\text{I}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.89</td>
<td>0.233</td>
</tr>
<tr>
<td>1.22</td>
<td>0.530</td>
</tr>
<tr>
<td>1.54</td>
<td>0.87</td>
</tr>
</tbody>
</table>

Special Equipment: Sealing and remotely operated opening devices for aluminum irradiation container. Remotely operated diamond cut-off wheel. Remotely operated sample transfer and chemical processing equipment, including jigs to position transfer container.
A. Target Preparation

1. Wrap 2 g \( \text{U}_3\text{O}_8 \) powder in aluminum foil.

2. Place in quartz ampoule and weld quartz closed.

3. Place in aluminum irradiation can and crimp on lid.

B. Post-Irradiation Handling

1. Pry the lid off the aluminum can. Use remote operation and shielding.

2. Transfer the quartz ampoule to a lathe chuck and, while rotating, cut end off quartz with a high speed diamond wheel.

3. Dump the foil-wrapped \( \text{U}_3\text{O}_8 \) into a transfer container and transfer to a dissolving vessel.

The dissolving vessel is a 500-ml round bottom flask resting in an electric heating mantle. It has a stopperable charging inlet, a stopcock-regulated reagent supply line, a gas bubbler, and a heated exhaust line leading to a water-cooled condenser.

C. Post-Irradiation Processing

1. Add 30 ml 12 N \( \text{H}_2\text{SO}_4 \) to the dissolving vessel.

2. Heat the dissolver and the line to the condenser.

The aluminum foil wrapping dissolves when the solution is heated.

3. Add a few drops of 1 N \( \text{NaOH} \) to the condenser discharge.

The condenser drains into a small (1 - 2 ml) vessel containing glass beads to insure good gas-liquid contact; into this vessel is led also a reagent addition funnel. The vessel drains into a collection vessel which automatically siphons when 4 ml is collected.

4. Admit \( \text{N}_2 \) into the dissolver via the gas bubbler.

This sweeps out volatilized product.
5. As each 4 ml of product is collected, measure radiation level and store. Repeat step #3 each time.

6. When the aluminum foil wrapping is completely dissolved add 30 ml H₂O slowly.

7. Add 25 ml 30% H₂O₂ 2 - 3 drops per second.

8. Turn off heat when 12 - 16 ml product have been collected.

9. Select most radioactive fraction for assay and shipping.

The first 4 ml usually contains only little activity.

The 4 ml collected during addition of the H₂O₂ contains the bulk of the activity.
Magnesium-28

Isotope: $\text{Mg}^{28}$

Half-Lives: $\text{Mg}^{28}$, 20.9 h; $\text{Al}^{27}$ (daughter), 2.3 m

Production Method: $\text{Li}^6(n,t)\text{He}^4$; $\text{Mg}^{26}(t,p)\text{Mg}^{28}$ (reactor)

Cross Section: 950 b for $\text{Li}^6(n,t)\text{He}^4$

Target: $\text{Li}^6\text{Mg}^{26}$ alloy

Target Weight: ~12 mg

Neutron Flux: $(1.2 - 1.3) \times 10^{13} \text{ n sec}^{-1} \text{ cm}^{-2}$

Irradiation Time: ~3 days

Reactor Yield: ~100 $\mu$C $\text{Mg}^{28}$-$\text{Al}^{27}$

Chemical Yield: ~90%

Specific Activity: ~40 mC $\text{Mg}^{28}$-$\text{Al}^{27}$ per gram Mg at time of shipment

Product Composition: $\text{Mg}^{28}\text{Cl}_2$ in dilute HCl containing <1% NaCl

Radiochemical Purity: 95% $\text{Mg}^{28}$-$\text{Al}^{27}$, 5% H$^2$, traces of Cu$^{64}$, Cd$^{115}$ as of 24 hours following processing

Method of Analysis: Well-type scintillation counter standardized against $4\pi$ $\beta$ counter

Radiations (MeV):

<table>
<thead>
<tr>
<th></th>
<th>Beta ($\text{Mg}^{28}$)</th>
<th>Gamma ($\text{Mg}^{28}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.42 (100%)</td>
<td>0.032 (96%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.40 (31%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.95 (29%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.35 (70%)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Beta ($\text{Al}^{27}$)</th>
<th>Gamma ($\text{Al}^{27}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2.87</td>
<td>1.78</td>
</tr>
</tbody>
</table>

Special Equipment: Low moisture, inert atmosphere glove box (''dry box'') with air lock. Remotely operated can opener. Processing glove box with ~1/2-inch lead shielding suitable for handling gross tritium contamination.
A. Target Preparation

1. Weigh out correct amounts of Li$^6$ and Mg$^{28}$ metals separately and rapidly on an analytical balance.

2. Rapidly transfer metal samples into dry box air lock and flush with dry argon.

3. Transfer metal samples from air lock into dry box proper.

4. Melt samples together in graphite crucible at ~650°F in furnace, stirring with a graphite rod.

5. Pour molten alloy into shallow graphite mold.

6. Place ingot under oil for transfer out of box.

7. Using a suitable small rolling mill, roll to 0.25 mm thickness.

8. Cut into suitable strips for irradiation.

9. Store under oil

B. Target Irradiation

1. Clean alloy foil with ethyl acetate, weigh, and insert in slits in 3/4-inch diameter graphite rod.

2. Place loaded rod in suitable aluminum can and weld shut.

3. Evacuate can via tube on cap and fill with helium.

Solvent removes oil. Rod acts as spacer, holder, and heat sink and has two longitudinal slits, cut about 3/4 of the way through. Dry box not needed if handling is fairly rapid.

Cap of can should have short length of 1/8-inch diameter tube attached to it.

Ordinary oil-pumped helium is satisfactory. Helium prevents air oxidation and is a good heat transfer agent.
4. Crimp tube closed.  
5. Irradiate in reactor ~3 days.  
6. Open can with remotely operated opener.

End need not be welded if crimp seal is a good one.  
Use cooled hole to prevent sample from melting. Note: If sample melts, thickness will increase and specific activity of Mg will decrease; see above.  
Enclosure must be capable of dealing with considerable tritium contamination from Li\(^{6}(n,\alpha)H\).

C. Post-Irradiation Processing

1. Transfer graphite rod and alloy foils to processing glove box.  
Dry inert atmosphere not required.

2. Remove foils from rod and dissolve, one at a time, in a minimum of \(H_2O\) in a 50-ml plastic centrifuge tube in an ice bath.  
Reaction is rapid. If foils are added too rapidly, they may ignite. Plastic centrifuge tubes insure against accidental breakage.

3. Add 3 drops \(3\ N\ NaOH\) and centrifuge.  
Assures minimum loss of Mg.  
Centrifuge 5 min.

4. Discard supernate.  
Contains \(F^1\), \(\alpha\) and \(Na^{24}\). \(F^1\) comes from \((t,\alpha)\) reaction on oxide impurities. \(\alpha\) comes from unreacted tritons. \(Na^{24}\) comes from \((n,\gamma)\) reaction on sodium impurities, from \((t,\alpha)\) reaction on residual Mg, and from \((n,\alpha)\) reaction on Al impurities.

5. Wash twice with ice water containing 3 drops of \(3\ N\ NaOH\), discarding supernate.  
Reduces \(H\) and \(Na^{24}\) contamination.

6. Dissolve \(Mg(OH)_2\) with conc. HCl.  
Use ~0.5 ml HCl/10 mg alloy.

7. Pour into 50-ml beaker containing stirring bar and place on magnetic mixer.  
Steps #7 - #10 are necessary only if original \(Mg^{26}\) became contaminated with Al during the reduction to metal.

8. Add 3 drops of 0.1% methyl red indicator while stirring.  
This precipitates \(Al(OH)_3\). The end point is critical. \(Mg(OH)_2\) will precipitate if pH goes too high.

9. Add 0.1 \(N\ NaOH\) until just yellow.
10. Centrifuge and wash precipitate with H₂O.
11. To supernate add 3 mg F⁻ carrier and 1 drop of 3 N HCl.
12. Pour supernate onto prepared ion exchange column.

Combine supernate and washes and save. Discard precipitate.

13. Wash column with 75 ml of 0.001 N HCl.

14. Combine effluents and transfer to distillation flask. Distill to near dryness.

Use KF for carrier, 30 mg F⁻ per ml.

15. Add ~50 ml 0.001 N HCl and distill to near dryness.

Column preparation: In a 1-inch fritted Buchner funnel settle 3/4 inch of Dowex 1 x 8 (Cl⁻ form). Cover with cloth disc and add 3/4 inch of Woelm Al₂O₃ (neutral grade) which had been previously conditioned with 0.5 N HCl and washed twice with H₂O. Column removes remainder of F⁻. This is necessary for subsequent Mg²⁺ assay.

16. Repeat step #15.

Use glass beads in flask to prevent bumping. Discard distillate. Steps #14 - #16 reduce volume of product (residual solution) and H⁺ contamination in it.

17. Transfer residual solution from distillation flask to beaker.

18. Wash distillation flask at least twice with 0.001 N HCl and combine with residual solution (step #17).

Final volume should be ~5 ml.
Solution should be water clear.
Molybdenum-99

Isotope: Mo\textsuperscript{99}

Half-Lives: Mo\textsuperscript{99}, 67 h; Tc\textsuperscript{99m} (daughter), 6.0 h

Production Method: \textsuperscript{235}U(n,f) (reactor)

Fission Cross Section: 582 b

Target: Alloy of Al with U which has been enriched to 93.15\% \textsuperscript{235}U

Target Weight: \textasciitilde 180 mg \textsuperscript{235}U; \textasciitilde 7 g Al

Neutron Flux: \textasciitilde 1.2 \times 10\textsuperscript{13} n sec\textsuperscript{-1} cm\textsuperscript{-2}

Irradiation Time: \textasciitilde 3 d

Fission Yield: \textasciitilde 6\%

Reactor Yield: \textasciitilde 2.3 C Mo\textsuperscript{99}

Chemical Yield: \textasciitilde 70\%

Specific Activity: "Carrier-free" Mo\textsuperscript{99} (i. e. no Mo carrier added)

Product Composition: (NH\textsubscript{4})\textsubscript{2}Mo\textsuperscript{99}O\textsubscript{4} in 1 M NH\textsubscript{4}OH

Radiochemical Purity: \textasciitilde 99.99\% Mo\textsuperscript{99}, traces of I\textsuperscript{131} and Ru\textsuperscript{106} (at time of shipment)

Method of Analysis: Well-type scintillation counter standardized against \gamma spectrometer

Radiations (MeV):

<table>
<thead>
<tr>
<th>Beta (Mo\textsuperscript{99})</th>
<th>Gamma (Mo\textsuperscript{99})</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.45 (14%)</td>
<td>0.041</td>
</tr>
<tr>
<td>0.37 (~1%)</td>
<td>0.181</td>
</tr>
<tr>
<td>1.23 (85%)</td>
<td>0.372</td>
</tr>
<tr>
<td></td>
<td>0.740</td>
</tr>
<tr>
<td></td>
<td>0.780</td>
</tr>
<tr>
<td></td>
<td>0.950</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Beta (Tc\textsuperscript{99m})</th>
<th>Gamma (Tc\textsuperscript{99m})</th>
</tr>
</thead>
<tbody>
<tr>
<td>\textless 0.03% primary $\beta^-$</td>
<td>0.002</td>
</tr>
<tr>
<td>9.5% internal conversion electrons</td>
<td>0.140</td>
</tr>
<tr>
<td></td>
<td>0.142</td>
</tr>
</tbody>
</table>
Special Equipment: Chemical processing cell providing 3 feet of normal concrete or 1 foot of steel shielding. Remotely operated chemical processing equipment. Shielded remotely operated can opener.

A. Target Preparation

1. Cut a piece of Al-U alloy to contain \( \sim 180 \text{ mg } U^{35} \), trimming off excess aluminum at edges.

2. Place alloy in suitable aluminum can and weld shut.

3. Evacuate can via tube on cap and fill with helium.

4. Crimp tube closed.

Rejected scrap fuel plate (originally intended for the BNL Graphite Research Reactor) is used. This is an alloy of Al-U sandwiched between two aluminum plates which extend \( \sim 1/4 \) inch beyond the alloy.

Cap of can should have short length of 1/8-inch-diameter tube attached to it.

Helium prevents air oxidation and is a good heat transfer agent.

End need not be welded if crimp seal is a good one.

B. Chromatographic Column Preparation

1. Wash 50 ml alumina with \( H_2O \), discarding fines.

2. Slurry the alumina into the column.

3. Slurry onto the alumina a layer of ground glass.

4. Wash the alumina with the following in this order:
   - 200 ml \( 1 \text{ M } HNO_3 \),
   - 100 ml \( H_2O \),
   - 100 ml \( 1 \text{ M } NH_4OH \),
   - 200 ml \( H_2O \),
   - 100 ml \( 1 \text{ M } HNO_3 \)

Use Fisher chromatographic alumina, 50 - 200 mesh.

See schematic diagram of equipment (Figure 11).

This keeps subsequently added reagents from disturbing the alumina bed.

This treatment artificially "ages" the alumina and reduces the amount of Al\(^{+++}\) leached off by subsequent processing.
C. Post-Irradiation Handling

1. Load irradiated can into a suitable portable transfer shield and transfer to can-opening cave on top of hot cell.

2. Open can remotely and drop irradiated target into the dissolver vessel in the cell.

See introductory section preceding these individual procedures for a description of this equipment and its operation.

D. Post-Irradiation Processing

1. To the region under the filter frit of the dissolver vessel apply sufficient air pressure (1 - 4 psig) to force a small flow of air through the frit.

2. Cover the irradiated target with 48 ml 6 M HNO₃ and 2 ml 2 M Hg(NO₃)₂.

3. Apply heat to start the dissolution. Turn off heat when the reaction becomes vigorous.

4. Cautiously add 3 more 50-ml portions of 6 M HNO₃, pausing between additions.

5. After solution is clear, add 5 ml Tc carrier solution (0.1 mg Tc/ml 5 M HNO₃).

6. Wash Tc carrier into dissolver with a few ml 6 M HNO₃.

7. Add H₂O to bring volume to ~500 ml.

8. By turning off the air pressure under the frit and applying air pressure above the frit as needed, drain the solution through the frit onto the alumina column.

This prevents any subsequently added liquid from draining through the frit.

If this volume fails to cover the target completely add enough 6 M HNO₃ to do so. The Hg(NO₃)₂ catalyzes the dissolution.

Caution! Reaction can become violent.

Caution! Reaction will run away if acid is added too rapidly.

It may be necessary to wait a few minutes for solution to clear before adding Tc; it may also be necessary to add an additional 50-ml portion of 6 M HNO₃ to clear the solution.

This avoids loss of carrier by hold-up in inlet lines.

Up to 15 psig air pressure may be used to maintain a liquid flow rate of 10 - 15 ml/min. If filter frit plugs up, back-blowing with air may be necessary to clear it.
9. Rinse dissolver vessel with 200 ml 1 M HNO₃ and filter onto alumina column.

10. Run 2 200-ml portions of 1 M HNO₃ onto alumina column. This washes unadsorbed ions off the alumina.

11. Run 100 ml H₂O onto alumina column, followed by 100 ml 0.01 M NH₄OH. This removes any traces of acid and makes the alumina slightly basic.

12. Run 145 ml 1 M NH₄OH onto alumina. Discard the first 20 ml of effluent and transfer the remainder to a mixing vessel and then to a dispensing buret. 1 M NH₄OH elutes Mo⁹⁹, but none appears in the first 20 ml.

Figure 11. Fission product separation equipment.
Potassium-43

Isotope: $^{43}\text{K}$

Half-Life: 22 h

Production Method: $^{40}\text{Ar} (\alpha, p)^{43}\text{K}$ (cyclotron)

Target: gaseous Ar; ~18-mil window

Target Area: ~1.5 x 6 inches

Target Thickness: 5 inches (along direction of beam)

Target Pressure: 15 psig

Beam Energy: ~40 MeV

Integrated Beam Current: 20 μAh

Cyclotron Yield: 1 mC $^{43}\text{K}$

Chemical Yield: 80 - 90% $^{43}\text{K}$

Specific Activity: Carrier-free

Product Composition: $^{43}\text{K}$ in $\text{H}_2\text{O}$, saline solution, etc. as required

Radiochemical Purity: ~10% $^{42}\text{K}$; remainder $^{43}\text{K}$ as of end of bombardment

Method of Analysis: Precalibrated well-type scintillation crystal

Radiations (MeV):

<table>
<thead>
<tr>
<th>Beta</th>
<th>Gamma</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.47 (8%)</td>
<td>0.22 (3%)</td>
</tr>
<tr>
<td>0.83 (87%)</td>
<td>0.37 (85%)</td>
</tr>
<tr>
<td>1.24 (3.5%)</td>
<td>0.39 (18%)</td>
</tr>
<tr>
<td>1.81 (1.5%)</td>
<td>0.59 (13%)</td>
</tr>
<tr>
<td>1.83 (2%)</td>
<td>0.61 (81%)</td>
</tr>
<tr>
<td>1.01 (2%)</td>
<td></td>
</tr>
</tbody>
</table>

A. Target Preparation

1. Evacuate target chamber (to 10 - 20 μ) and fill with argon.

2. Repeat step #1 several times.

3. Fill with argon to 15 psig.

Target chamber is an aluminum can with an 18-mil window on one 6 x 1.5 inch face; the third dimension (in beam direction) is 5 inches; two needle valves permit flushing and draining. Window is intentionally thick to withstand pressure difference and to degrade energy to minimize competing Ar40(α,pn)Kr42 reaction.

B. Post-Irradiation Processing

1. Open the top valve on the target chamber, releasing the argon in a hood.

2. Through a funnel inserted into the upper valve, add 10 ml suitable rinse solution.

3. Close both valves. Shake and rotate chamber so that all inner surfaces are wet.

4. Drain solution through bottom valve into collection bottle.

5. Repeat steps #1 - #4.

Note: Rinse target chamber out thoroughly to prevent corrosion prior to re-use.
**Scandium-48**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isotope:</td>
<td>Sc$^{48}$</td>
</tr>
<tr>
<td>Half-Life:</td>
<td>44 h</td>
</tr>
<tr>
<td>Production Method:</td>
<td>$V_2^6\text{(n,}\alpha)\text{Sc}^{48}$ (Cockroft-Walton generators)</td>
</tr>
<tr>
<td>Production Cross Section:</td>
<td>18 mb</td>
</tr>
<tr>
<td>Target:</td>
<td>$V_2\text{O}_5$</td>
</tr>
<tr>
<td>Target Weight:</td>
<td>100 g</td>
</tr>
<tr>
<td>Neutron Flux:</td>
<td>$\sim5 \times 10^8$ n sec$^{-1}$ cm$^{-2}$</td>
</tr>
<tr>
<td>Neutron Source:</td>
<td>$H^0(d,\text{n})\text{He}^4$</td>
</tr>
<tr>
<td>Beam Energy:</td>
<td>Fast neutrons are required; 14 - 15 MeV neutrons are produced from $\sim150$ keV deuterons.</td>
</tr>
<tr>
<td>Integrated Beam Current:</td>
<td>$\sim30 \mu$Ah of deuterons</td>
</tr>
<tr>
<td>Accelerator Yield:</td>
<td>$\sim2 \mu$C</td>
</tr>
<tr>
<td>Chemical Yield:</td>
<td>50%</td>
</tr>
<tr>
<td>Specific Activity:</td>
<td>Carrier-free</td>
</tr>
<tr>
<td>Product Composition:</td>
<td>Sc$^{48}\text{Cl}_3$ (&quot;weightless&quot; solid)</td>
</tr>
<tr>
<td>Radiochemical Purity:</td>
<td>$&gt;99.3%$</td>
</tr>
<tr>
<td>Method of Analysis:</td>
<td>Gamma spectroscopy and decay of 175 keV $\gamma$</td>
</tr>
<tr>
<td>Radiations (MeV):</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Beta</td>
</tr>
<tr>
<td></td>
<td>0.65 (93%)</td>
</tr>
<tr>
<td></td>
<td>0.48 (7%)</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>
A. Target Preparation and Irradiation

1. Place bulk target material in a plastic bag and suspend in the path of the fast neutrons.

Neutrons are generated by deuteron bombardment of $^3H$ in the form of the tritide of Y, Ti, or Zr. Neutron yield depends as much on quality of target as on deuteron beam current.

B. Post-Irradiation Processing

1. Cover the $V_2O_5$ with 150 ml $H_2O$ and dissolve by adding 150 ml hot NaOH solution with stirring.

Use 80 g NaOH in 150 ml $H_2O$.

2. Dilute to 1000 ml.

Separation of a red solid should be avoided although a small amount is not troublesome.

3. With stirring pour slowly into 2000 ml 2 M HCl.

Fe(OH)$_3$ precipitates, carrying $Sc^{4+}$ and some V.

4. Add 10 ml 1 M FeCl$_3$.

Concentrations are unimportant, but volumes should be kept small. This step removes the remaining V.

5. With stirring add slowly 1000 ml 4 M NaOH.

6. Centrifuge, discarding supernate.

7. Dissolve precipitate in HCl and reprecipitate with NaOH.

8. Repeat steps #6 - #7 twice.

9. Dissolve precipitate in 100 ml 8 M HCl.

10. Extract with 100 ml diethyl ether.

Use ether which has been pre-equilibrated with 8 M HCl.

11. Partially neutralize aqueous layer with conc. NH$_4$OH.

The solution gets hot, the residual ether boils off, and some $Sc^{4+}$ is lost.

12. Add 11 mg Fe$^{3+}$ carrier and reprecipitate with NH$_4$OH.

If any tetravalent V is present, a blue precipitate, insoluble in acid, will appear. To remove it, centrifuge the solution while acid. No $Sc^{4+}$ is lost on this precipitate.

13. Repeat steps #6 - #7 several times, substituting NH$_4$OH for NaOH.
14. Repeat step #9 using ~10 ml 8 M HCl and repeat step #10 several times, using ~10 ml ether.

This removes the remaining Fe.

15. Raise pH of aqueous layer to 3.

16. Extract with 10 ml of a solution of 0.5 M TTA in benzene. Discard aqueous layer.

TTA is the common designation for thenoyltrifluoroacetone.

17. Wash benzene layer several times with distilled H₂O, discarding washes.

This removes salts (viz. NH₄Cl) carried in the benzene.

18. Extract benzene twice with 2 M HCl, saving aqueous extract.

19. Wash aqueous layer several times with benzene.

This removes TTA carried by the water.

20. Evaporate aqueous layer to dryness.

No solids should appear.

Note: Thus far, no effort has been made to find optimum conditions for high chemical yield. Although not yet tried, a superior procedure to steps #15 - #19 might be to bring the solution to 10 M in HCl by adding 20 - 30 ml conc. HCl, extracting Sc⁴⁺ into tri-n-butyl phosphate, and back extracting with H₂O which is then washed with ether.
**Strontium-87m**

**Isotope:** Sr\(^{87m}\)

**Shipping Form:** Generator

**Parent:** Y\(^{87}\)

**Half-Lives:** Sr\(^{87m}\), 2.8 h; Y\(^{87}\), 80 h

**Production Method:** Rb\(^{85}\) (\(\alpha,2n\)) Y\(^{87}\) (cyclotron)

**Target:** RbCl (unenriched); 5-mil aluminum window

**Target Thickness:** ~0.02 inch

**Beam Energy:** ~40 MeV

**Integrated Beam Current:** ~15 \(\mu\)Ah

**Cyclotron Yield:** >100 \(\mu\)C Y\(^{87}\)/\(\mu\)Ah

**Chemical Yield:** >90% Y\(^{87}\)

**Generator Yield:** ~70% Sr\(^{87m}\)

**Specific Activity:** "Carrier-free" Sr\(^{87m}\) (i.e. no Sr carrier added)

**Product Composition:** (Sr\(^{87m}\))\(_3\) (C\(_6\)H\(_5\)O\(_7\))\(_2\) in 0.005% citric acid (aq.)

**Radiochemical Purity:** >99.9% at time of milking

**Method of Analysis:** Well-type scintillation counter standardized against gamma spectrometer

**Radiations (MeV):**

<table>
<thead>
<tr>
<th>Type</th>
<th>Isotope</th>
<th>Energy (MeV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beta (Sr(^{87m}))</td>
<td>none</td>
<td>none</td>
</tr>
<tr>
<td>Beta (Y(^{87}))</td>
<td>0.3% (\beta^+)</td>
<td>0.39</td>
</tr>
<tr>
<td>Gamma (Sr(^{87m}))</td>
<td></td>
<td>0.483</td>
</tr>
<tr>
<td>Gamma (Y(^{87}))</td>
<td></td>
<td>0.483</td>
</tr>
</tbody>
</table>

**Special Equipment:** Inert atmosphere box for preparing target. Shielded remotely operated target dismantling device. 2 inches of lead shielding locally around processing equipment.
A. Target Preparation

1. Place ~4.25 g dry RbCl powder in a 0.05" x 0.75" x 5" recess in a copper strip. Copper is chosen for good heat transfer during bombardment.


3. Cover RbCl with a 5-mil aluminum window and fasten copper strip to a water-cooled aluminum target holder.

B. Preparation of Generator

1. Place ~1/8 inch finely ground quartz on the frit of a 25 mm x 80 mm Büchner funnel. Do not disturb quartz.

2. Add 25 ml Dowex 1 x 8 resin (50 - 100 mesh).

3. Cover resin with cloth, cover cloth with 1/8 inch quartz, cover quartz with cloth, and add a polyethylene retainer ring. The quartz controls the flow rate to the desired 1 - 2 ml/min. The cloth should be woven from a non-adsorbing synthetic fiber; "Feon", an acrylic filter cloth, is suitable. The spring action of the retainer ring holds it and the material underneath in place.

4. Wash resin with H₂O until no Cl⁻ is detectable. Requires 100 - 200 ml H₂O. Use AgNO₃ solution to detect Cl⁻.

5. Pass 200 ml 0.5 N NaHCO₃ solution through column, followed by 200 ml H₂O. This puts column in HCO₃⁻ form.

C. Post-Irradiation Processing of Parent Yb⁷⁺

1. Remove target from aluminum holder; transfer to processing equipment. Use remotely operated target dismantling device.

2. Place target (copper strip and RbCl) in special vessel and cover with ~18 ml water. Target rests on its side in Lucite vessel provided with stopcock for draining.
3. After RbCl is dissolved, filter solution into 50-ml centrifuge tube.

4. Cover copper target strip with diluted Y carrier solution.

5. Filter solution into centrifuge tube, rinsing Lucite vessel with H₂O and filtering rinse into centrifuge tube.

6. Remove copper strip from work area.

7. Add 20 drops conc. NH₄OH to solution in centrifuge with swirling.

8. Centrifuge, discarding supernate.

9. Dissolve precipitate with 6 drops 6 N HCl.

10. Reprecipitate with 6 drops conc. NH₄OH.

11. Repeat step #8.

12. Add 1 ml H₂O to precipitate; then dissolve by adding 3 - 6 drops 3 N HCl.

13. Add ~10 ml H₂O and 10 ml 0.5% citric acid solution. Mix.


15. Transfer solution to dispensing burette.

D. Loading the Generator

1. Drip solution from step #15, section C, onto resin column prepared in section B.

Use coarse Buchner funnel to remove particles of copper corrosion.

Diluted carrier solution: 17 ml H₂O + 1 drop 6 N HCl + 1 ml Y carrier (3.5 mg Y/ml).

This will reduce radiation level.

Y(OH)₃ precipitates.

~5 min is sufficient.

Solution should be clear.

pH of citric acid solution should be 5.5.
2. Wash resin with 50 - 100 ml 0.005% citric acid solution. Use fresh citric acid solution each time; adjust pH to 5.0 just before using.

Note: Generator is milked (i.e. Sr$^{90}$m is eluted from resin) with 20 ml 0.005% citric acid solution, the pH of which has been adjusted to 5.0.
Technetium-99m

Isotope: Tc$^{99m}$
Shipping Form: Generator
Parent: Mo$^{99}$
Half-Lives: Tc$^{99m}$, 6.0 h; Mo$^{99}$, 67 h
Production Method: See Molybdenum-99
Generator Yield: ~70%
Specific Activity: Carrier-free
Product Composition: (Tc$^{99m}$Mo$_4$)$^-$ in 0.1 M HCl
Radiochemical Purity: >99.9% Tc$^{99m}$ at time of milking; traces of Tl$^{31}$ and Ru$^{103}$
Method of Analysis: Well-type scintillation counter standardized against $\gamma$ spectrometer

Radiations (MeV):

<table>
<thead>
<tr>
<th>Radiation Type</th>
<th>Energy (MeV)</th>
<th>Activity (cpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beta (Tc$^{99m}$)</td>
<td>&lt;0.03% primary $\beta^-$</td>
<td>0.002</td>
</tr>
<tr>
<td></td>
<td>9.5% internal conversion electrons</td>
<td>0.140</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.142</td>
</tr>
<tr>
<td>Beta (Mo$^{99}$)</td>
<td>0.45 (14%)</td>
<td>0.041</td>
</tr>
<tr>
<td></td>
<td>0.37 (~12%)</td>
<td>0.181</td>
</tr>
<tr>
<td></td>
<td>1.23 (85%)</td>
<td>0.372</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.740</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.780</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.950</td>
</tr>
</tbody>
</table>

Special Equipment: Remotely operated dispensing equipment. $\frac{1}{2}$-inch-thick lead shield to enclose generator during shipping.

---

A. Preparation of Generator

1. Wash 5 g Woelm (neutral grade) alumina with 0.1 M HNO$_3$, decanting and discarding fines. Continue washing until effluent is strongly acid (pH = 1 - 2).
2. Load a glass Buchner funnel with the alumina.

3. Cover with Feon cloth, held down by a polyethylene retainer ring.

4. Wash with 25 ml 0.1 M HNO₃.

B. Loading the Generator

1. Into a few ml H₂O pipet the required amount of Mo⁹⁹ product solution.

2. Add several drops Crystal Violet indicator solution.

3. With stirring, add HNO₃ to indicator endpoint (pH = 1 - 2).

4. With stirring, add H₂O to bring volume to ~25 ml.

5. Drip into generator (see above).

6. Pass 50 ml 0.1 M HCl through generator.

Note: Generator is milked (i.e., Tc⁹⁹m is eluted from alumina) with 15 - 25 ml 0.1 M HCl.
Tellurium-132

Isotope: Te\(^{132}\)

Half-Lives: Te\(^{132}\), 77 h; I\(^{132}\) (daughter), 2.3 h

Production Method: \(^{90}\)Sr\(^{(n,f)}\)Te\(^{132}\) + ... (reactor)

Fission Cross Section: 582 b

Target: Alloy of Al with U which has been enriched to 93.15\% \(^{235}U\)

Target Weight: \(-180\) mg \(^{235}U\); \(-7\) g Al

Neutron Flux: \(-1.2 \times 10^{13}\) n sec\(^{-1}\) cm\(^{-2}\)

Irradiation Time: \(-3\) d

Fission Yield: 4.7\%

Reactor Yield: \(-1.9\) C Te\(^{132}\)

Chemical Yield: 50\%

Specific Activity: \(-500\) C/g Te 2 d after end of irradiation

Product Composition: \(H_2Te^{132}O_3\) in 1 M HNO\(_3\)

Radiochemical Purity: \(A_{Te^{131}}/A_{Te^{132}} = 15.9\%\) at end of irradiation, and 8\% 2 days later, etc. Ratio decreases with a half-life of 49.2 hours.

Method of Analysis: Well-type scintillation counter standardized against \(^{40}\)Ca counter

Radiations (MeV):

<table>
<thead>
<tr>
<th>Beta (Te(^{132}))</th>
<th>Gamma (Te(^{132}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22</td>
<td>0.052</td>
</tr>
<tr>
<td></td>
<td>0.233</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Beta (I(^{132}))</th>
<th>Gamma (I(^{132}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.80 (21 %)</td>
<td>0.528</td>
</tr>
<tr>
<td>1.04 (15 %)</td>
<td>0.670</td>
</tr>
<tr>
<td>1.22 (12 %)</td>
<td>0.777</td>
</tr>
<tr>
<td>1.49 (12 %)</td>
<td>0.96</td>
</tr>
<tr>
<td>1.61 (21 %)</td>
<td>and &gt;23 other (\gamma)'s from 0.240</td>
</tr>
<tr>
<td>2.14 (18 %)</td>
<td>to 2.9 MeV</td>
</tr>
<tr>
<td>2.92 (0.03%)</td>
<td></td>
</tr>
</tbody>
</table>

- 55 -
Special Equipment: Chemical processing cell providing 3 feet of normal concrete or 1 foot of steel shielding. Remotely operated chemical processing equipment. Shielded remotely operated can opener.

Note: For Target Preparation, Chromatographic Column Preparation, Post-Irradiation Handling, and first 12 steps of Post-Irradiation Processing, see procedure for Molybdenum-99.

D. Post-Irradiation Processing (continued)

13. Run 400 ml conc. NH₄OH onto alumina column, discarding effluent.

14. Run 50 ml 0.01 M NH₄OH onto alumina column, discarding effluent.

15. Run 95 ml 3 M NaOH onto alumina column, discarding the first 15 ml of effluent and transferring the remainder to the Te¹³² product collection vessel.

16. Wash alumina with 20 ml H₂O.

17. Transfer Te¹³² product from collection vessel to repurification vessel, maintaining air pressure under filter frit in latter vessel.

18. To product in repurification vessel add 60 ml conc. HCl containing 1 mg Te carrier.

19. Switch from compressed air to gaseous SO₂ under filter frit, allowing SO₂ to bubble through solution above frit until precipitation is complete.

20. Switch from compressed SO₂ to partial vacuum under filter frit, filtering Te, and diverting filtrate to waste.

This removes most of any residual Mo⁹⁹ left on the alumina.

Combine wash with Te¹³² product from preceding step.

Repurification vessel is smaller but similar in principle to dissolver vessel, having filter frit at bottom; it and connecting lines are not shown in Figure 11. Air pressure under frit keeps solution from entering frit.

SO₂ reduces Te to the element (black precipitates). Complete precipitation requires ~5 min.

Use only as much vacuum as needed (250 - 400 torr).
21. Readmit \( \text{SO}_2 \) gas under filter frit. Then add 100 ml \( \text{H}_2\text{O} \), allowing \( \text{SO}_2 \) to bubble through it.

22. Repeat steps #20, #21, and #20.

23. Apply compressed air under filter frit and add 5 ml 10 N \( \text{HNO}_3 \).

24. When no more Te is visible, add 5 ml \( \text{H}_2\text{O} \) and wait 5 min.

25. Add 40 ml \( \text{H}_2\text{O} \) and store or load \( ^{132}\text{Tl} \) generator (see Iodine-132). Alternatively, go to step #25a.

25a. Make 1 M NaOH before shipping solution per se.

This washes the Te precipitate. Te is less soluble in water saturated with \( \text{SO}_2 \) than in pure \( \text{H}_2\text{O} \).

This will dissolve Te.

This will dissolve any traces of Te not dissolved in step #23.

Solution is now 1 N \( \text{HNO}_3 \) and is ready for loading \( ^{132}\text{Tl} \) generators and for storage. Acid condition is required for storage to prevent Te from precipitating on standing. Storage must be in continuously running hood to accommodate radio-iodine emanation.

This prevents emanation of radio-iodine in transit. Any precipitated Te can be redissolved again in \( \text{HNO}_3 \) at destination.
Xenon-128

Isotope: \( \text{Xe}^{128} \)

Shipping Form: Gas sealed in glass ampoule

Half-Life: \( \infty \) (stable)

Production Method: 
\[
\text{I}^{137}(n,\gamma)\text{I}^{128} \xrightarrow{\beta^-} \text{Xe}^{128}
\]

Production Cross Section: 5.6 b

Target: KI crystals, optical grade, grown under He atmosphere, and wrapped in Al foil

Target Weight: 100 g

Neutron Flux: \( 10^{13} \) n sec\(^{-1}\) cm\(^{-2}\)

Irradiation Time: 65 h

Reactor Yield: \( \) Chemical Yield: 0.15 cc (STP) = overall yield

Product Composition: \( \gg 95\% \text{ Xe}^{128} \)

Radiochemical Purity: No radioactivity present (see item \#4 below)

Method of Analysis: Mass spectrograph

Special Equipment: Same as for Ar\(^{38} \), which see

---

A. Post-Irradiation Processing

Note: Equipment and procedure are the same as for argon-38, which see, except for the following changes:

1. Wait for K\(^{42}\) to decay before processing.

2. In step \#9 of the Ar\(^{38}\) process, instead of bringing the charcoal up to room temperature, bring it up to 200°C in order to desorb Xe during gettering (which would not have occurred otherwise). Similarly in step \#13 and step \#16 the charcoal traps should be brought to 200°C. After step \#16, the charcoal should be allowed to return to room temperature before measuring the pressure; little, if any Xe, will be lost by readsoption.
3. In steps #7, #9, #11, #14, and #16, substitute dry ice-solvent for liquid N₂, because Xe does not require as low a temperature as Ar for adsorption.

4. Some 8.9-day Xeₑ₉⁹m is also formed by Xeₑ₉⁸(n,γ)Xeₑ₉⁹m, but the radioactivity can be reduced to any desired level by a sufficiently long cooling period.
Yttrium-90

Isotope: \( Y^{90} \)

Shipping Form: Generator

Parent: \( Sr^{90} \)

Half-Lives: \( Y^{90}, 64.2 \) h; \( Sr^{90}, 28 \) y

Production Method: \( ^{83}B(n,f)Sr^{90} + \ldots; \) (see ORNL Isotope Sales Department for details)

Generator Yield: 98%

Specific Activity: Carrier-free

Product Composition: ~0.98 mC \( Y^{90} \)/ml 0.5% citric acid solution (aq.) at radioactive equilibrium; proportionately less activity prior to equilibrium.

Radiochemical Purity: <10\(^{-3}\) mC \( Sr^{90} \)/ml; (note, therefore, that at radioactive equilibrium \( Sr^{90} \) is <10\(^{-5}\)% of \( Y^{90} \)).

Method of Analysis: Chemical separation and \( \beta^- \) counting \( Sr^{90} \); rapid assay possible prior to use of milked \( Y^{90} \) (described below).

Radiations (MeV): 

<table>
<thead>
<tr>
<th>Type</th>
<th>( Y^{90} )</th>
<th>( Sr^{90} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beta</td>
<td>2.26</td>
<td>0.54</td>
</tr>
<tr>
<td>Gamma</td>
<td>None</td>
<td>None</td>
</tr>
</tbody>
</table>

Special Equipment: Generator (see Figure 12 and description). Glove box with filters on inlet and exhaust ventilation lines and with partition separating relatively clean loading area from more contaminated storage area. Local shielding (against Bremsstrahlung) around \( Sr^{90} \) stock solution vessel. Assay column, ion exchange resin, carriers as described.
A. Resin Preparation

1. Place 200 ml Dowex 50W x 8 cation exchange resin on sintered glass filter disc in a glass column equipped with drain and overflow.

2. Wash resin with 200 ml H₂O flowing upward at ~4 ml/min.

3. Pass 800 ml 2 N NH₄Cl over resin at ~2 ml/min.

4. Pass 1000 ml H₂O over resin.

Use 50 - 100 mesh resin.

This removes excess H⁺ from resin. Upward flow is more rapid than downward.

This converts resin to NH₄⁺ form.

This removes excess NH₄Cl.

B. Generator Assembly

1. Load resin into specially built generator, leaving ~5 ml free space over main section and ~2 ml free space over top section.

2. Cap off both resin fill tubes and vent, leaving vessel entirely filled with liquid.

3. Connect product delivery tube to stopcock via Tygon connections.

4. Place generator in Lucite container. Connect generator outlet to stopcock via Tygon connection.

5. Attach bottom Lucite cover.

6. Place shock absorbing spacer between generator and Lucite.

7. Screw down top Lucite cover to seal against gasket.

See Figures 12 and 13. Spaces are free of resin but filled with liquid and permit expansion of resin during chemical loading and washing.

For fill tubes use small rubber "multi-injection" serum bottle stoppers. For vent use Tygon sleeve.

Delivery tube should swivel freely and retract when sliding cover closes.

Use a length of flexible plastic tubing compressed to fit.

Maintaining close tolerances in fabrication of generator will prevent breakage when cover is tightened.
C. Citric Acid Preparation

1. Weigh out 5.0 g anhydrous citric acid and 1.0 g phenol per liter of solution to be made.

2. Dissolve in required amount of distilled H₂O.

3. Set aside ~5% of solution in a separate container.

4. Using conc. NH₄OH, raise pH of main portion of citric acid solution to 5.4 - 5.5.

5. Add the 5% portion to the main portion of citric acid solution and readjust pH to 5.4 - 5.5 as in step #4.

D. Chemical Loading of Generator

1. Adjust flow rate through generator to ~3 ml/min.

2. Place generator in "clean" compartment of glove box, lining up eluent addition port with hole in ceiling of compartment.

3. Insert funnel through hole in compartment ceiling into eluent addition port and place suitable drain tube under product delivery tube.

4. Open generator stopcock and drain any excess liquid present.

An entire loading requires ≥2000 ml. Phenol prevents mold growth. Autoclaving of final solution may be substituted for phenol if pH is checked after autoclaving.

Save for "back-titrating" in case of overshooting pH.

Use accurate pH meter calibrated in this range. pH is critical and must be closely controlled and occasionally checked, if stored. Wrong pH will allow Sr⁹⁰ to migrate, eventually ruining generator.

See below for analysis to be performed prior to shipment.

Stopcock has screwdriver-operated Teflon needle valve for permanently setting desired flow rate when cock is full open.

Compartment isolates generator from Sr⁹⁰ storage and dispensing equipment to prevent surface contamination of generator.
5. Add required amount of $\text{Sr}^{90}$ stock solution slowly.

6. Wash funnel and generator with 3 30-ml portions of $\text{H}_2\text{O}$, allowing generator to drain after each washing.

7. Wash funnel and generator with 2 50-ml portions of citric acid preparation, allowing generator to drain after each washing.


9. Add enough powdered quartz (50 - 100 mesh) to fill re-entrant well over resin. Use citric acid preparation to wash quartz into place.

10. Check flow rate of generator. If necessary readjust to $\sim 3$ ml/min.

11. Pass 2000 ml citric acid preparation through the generator.

E. Milking Generator

1. Pour 100 ml citric acid preparation into top of generator through funnel.

Use dispensing buret. Prevent solution from approaching gasket.
Note: If $\leq 100$ mC $\text{Sr}^{90}$ are loaded, generator life will be $\sim 1$ year (before $\text{Sr}^{90}$ contamination in product begins to increase); if $>100$ mC are used, life will be shortened, presumably due to radiation damage of resin.

Radiation level at one foot from generator will be $\sim 6$ mR/hr for 100 mC loading.

Capillary action of quartz particles acts as a valve; when liquid level reaches quartz, flow stops automatically and air is prevented from entering generator even with stopcock still open. Air reaching the resin would cause subsequent channeling and disruption of the bed.

The citric acid reservoir is an orificed Torricellian tube arranged to provide a constant liquid flow and to shut off if the flow through the generator slows down or stops.

This will milk off whatever $\text{Y}^{90}$ has grown in since last milking. Knowing initial $\text{Sr}^{90}$ activity, age of generator, and time since last milking, consult graph (Figure 14) to see how much $\text{Y}^{90}$ is available.
2. Place 125 ml flask (see note 4) under delivery tube and open stopcock. [Flask should be shielded by ~0.5 inch plastic or wood.]

F. Pre-shipment Analysis for Sr\(^{90}\)

1. To 100 ml effluent from the generator add a known amount (~10 mg) Sr\(^{+}\) carrier as (~0.1 M) Sr(NO\(_3\))\(_2\). Mix well. [Caution: Observe proper precautions for handling ~100 mC \(^{90}\)Sr activity (\(\gamma^{90}\)).]

2. Add 10 ml saturated Na\(_2\)CO\(_3\), 5 ml conc. NH\(_4\)OH, and 1 - 2 drops phenolphthalein. [SrCO\(_3\) should precipitate. If solution turns colorless add more NH\(_4\)OH.]

3. Heat to near boiling for 30 min. with occasional stirring. [Discard supernates.]

4. Cool in ice bath for 30 min. [Use steam bath.]

5. Centrifuge, decant, wash precipitate with distilled H\(_2\)O, centrifuge, and decant. [Use distilled H\(_2\)O.]

6. Dissolve precipitate in 5 ml 1 N HCl and heat for 15 min. [\(^{99,90}\) (OH)\(_3\) precipitates.]

7. Cool and add 1 - 2 drops phenolphthalein and 3 - 4 mg \(^{99,90}\) carrier. [If solution is still pink, add HCl dropwise until colorless.]

8. Titrate with 1 N NaOH to first sign of pink color. [Decant supernate into 3 - 4 mg \(^{99,90}\) carrier and 1 - 2 drops 1 N HCl. Mix well.]

9. Make up volume to ~20 ml. [Repeat steps #8 - #11 three times. Then repeat steps #8 - 10.]

10. Let stand 5 - 10 min. with occasional shaking. Centrifuge. [Decant supernate into 3 - 4 mg Fe\(^{+}\) carrier and 1 - 2 drops 1 N HCl. Mix well.]

11. Decant supernate into 3 - 4 mg \(^{99,90}\) carrier and 1 - 2 drops 1 N HCl. Mix well. [Decant supernate into 3 - 4 mg Fe\(^{+}\) carrier and 1 - 2 drops 1 N HCl. Mix well.]

12. Repeat steps #8 - #11 three times. Then repeat steps #8 - 10.
4. Titrate with 1 N NaOH and add 2 drops excess.


16. To decanted supernate add 10 ml saturated oxalic acid solution and heat to near boiling.

17. While hot, add conc. NH₄OH drop-wise with stirring until a precipitate just forms. Digest hot for 15 min. with occasional stirring.

18. Cool in ice bath for 15 min. with occasional stirring.

19. Filter onto pre-weighed filter paper.

20. Wash precipitate with distilled H₂O, then with two 5-ml portions ethyl alcohol, then with two 5-ml portions ethyl ether. Suck dry.

21. Place filter paper and precipitate in vacuum desiccator and evacuate for 2 - 3 min.

22. Slowly admit air to desiccator.

23. Repeat steps #21 - #22.

24. Weigh and calculate percent recovery of Sr²⁺.

25. Mount precipitate and count β⁻⁻'s, with and without 215 - 220 mg/cm² aluminum absorber.

26. Correct count through the absorber for attenuation of Y⁹⁰ β⁻⁻'s by the absorber.

SrC₃O₄·H₂O (strontium oxalate monohydrate) precipitates.

Use ~15 mm diameter paper on a Hirsch funnel.

Use no desiccant.

This absorber removes all Sr⁹⁰ β⁻⁻'s, leaving only the background from residual Y⁹⁰.

Attenuation factor must be determined under identical conditions (backing, cover, thickness, area, position in counter) used for count in step #25.
Figure 12. Yttrium-90 generator.
Figure 13. Operation of yttrium-90 generator.

Figure 14. Curve showing growth of $\text{Y}^{90}$ from its $\text{Sr}^{90}$ parent and the effects of milking 2 weeks after and 1 day after previous milkings.
Figure 15. Strontium-90 assay column.
V. Subtract corrected count through absorber from count without absorber and correct for geometry and chemical yield.

This gives Sr\(^{90}\) activity per 100 ml product solution. (Note: Sr\(^{90}\) geometry correction must also be determined under conditions identical to those in step #25.)

G. Preparation and Use of Assay Column (by Customer)

1. Fill assay column to red line with resin.

A small plastic shielded glass column, Sr\(^{4+}\) carrier \([-0.1 \text{M } \text{Sr(NO}_3\text{)}_2]\), and Dowex 1 resin are furnished with each generator. See photograph (Figure 15). Raise or lower delivery tube as required.

2. Wash resin with 10 ml H\(_2\)O and adjust flow rate to \(\sim 2 \text{ ml/min.}\)

3. Add 1 ml Sr\(^{4+}\) carrier to a 5-ml aliquot of the Y\(^{90}\) product to be assayed, and mix.

4. Pour mixture into column, catching effluent in 25 ml volumetric flask.

5. Wash with \(10^{-3} \text{ M NH}_4\text{OH until } 25 \text{ ml effluent is collected.}\)

6. Pipet 2 ml samples onto each of 2 planchets.

7. Dry under heat lamp.

8. Count in duplicate as in steps #25 - #27 in section F above.

Note: This column assay procedure provides a simple rapid method whereby the customer can verify the purity of each milking before the batch is used. Sensitivity of this procedure is 0.01 - 0.02 \(\mu\text{C } \text{Sr}\(^{90}\). Although this is a factor of 10 higher than what should be present normally, it is still sufficiently sensitive for complete safety. If the procedure detects any Sr\(^{90}\) which is confirmed by repetition using a fresh aliquot of product, the generator should be returned to Brookhaven.