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MANUAL OF ISOTOPE PRODUCTION PROCESSES IN USE AT BROOKHAVEN NATIONAL LABORATORY

LOUIS G. STANG, JR., COORDINATOR

August 1964

BROOKHAVEN NATIONAL LABORATORY
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DISCLAIMER

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MANUAL OF ISOTOPE PRODUCTION PROCESSES IN USE AT BROOKHAVEN NATIONAL LABORATORY

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August 1964

HOT LABORATORY DIVISION
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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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CONTENTS

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 Sc^{48} all the isotopes described in this report are produced by irradiation either in a thermal neutron reactor or in a cyclotron; a Cockroft-Walton generator is used to make the fast neutrons needed for producing Sc^{48}. 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 μA or internal beam currents of ~300 μA. (In practice many targets present cooling problems which require limitation of beams to ~50 μA.)

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

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*See Graphite Research Reactor, Facilities and Services Guide, Revised July 1960, Brookhaven National Laboratory (brochure).

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^{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.

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*Stang, L. G. Jr., Precipitation Process and Apparatus Therefor, U. S. Patent No. 2533149.

**Stang, L. G. Jr., Tucker, W. D., Rand, A. C. Jr., Strickland, G., and Selvin, G. Remotely Operated Chemical Processing Equipment, BNL 8064; Stang, L. G. Jr., Experience With Brookhaven Hot Cells, ANL 4670, pp. 152-165; Stang, L. G. Jr., Radioisotope Facilities Associated With a Research Reactor, BNL 3227.

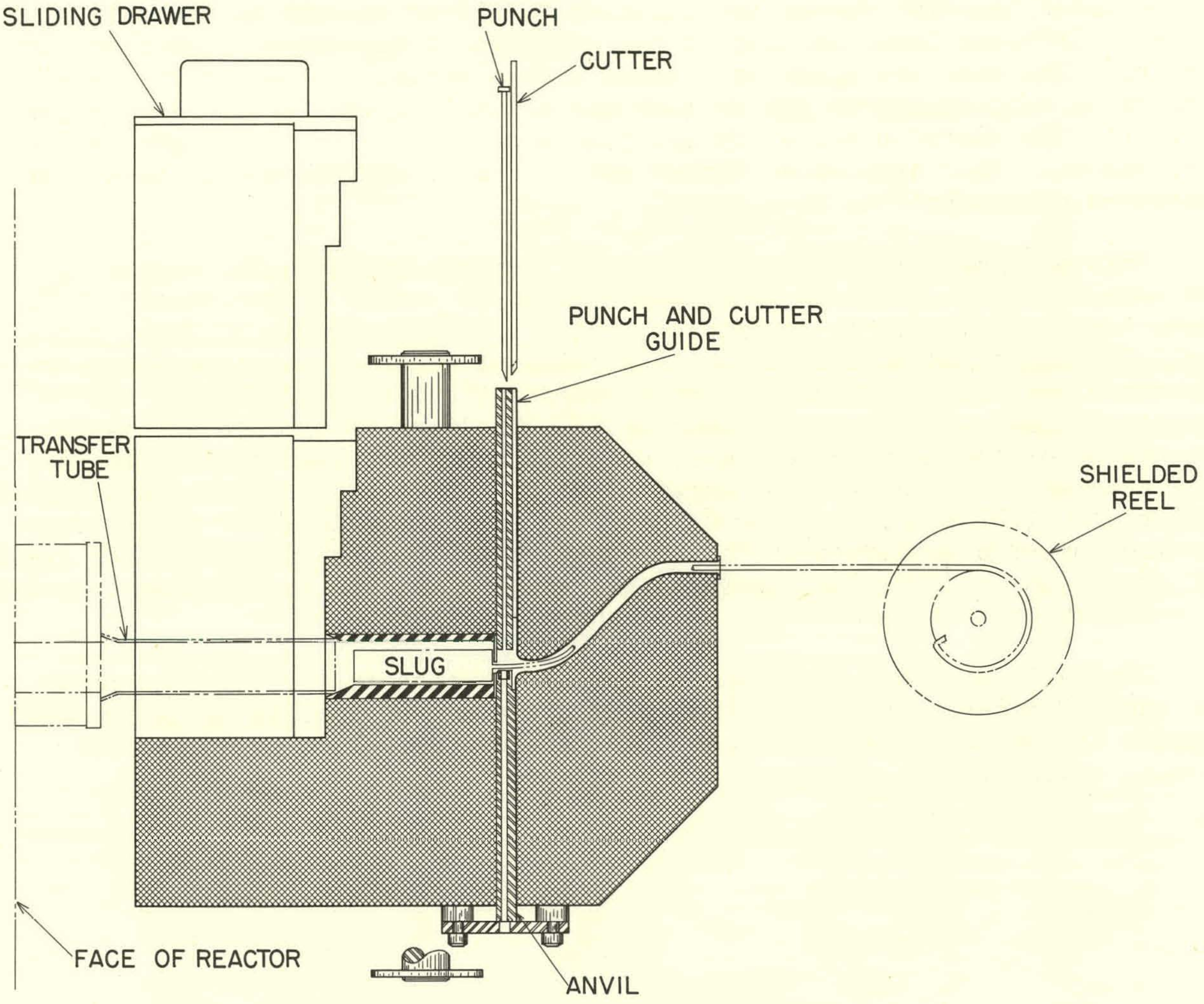

Figure 1. Slug transfer shield.

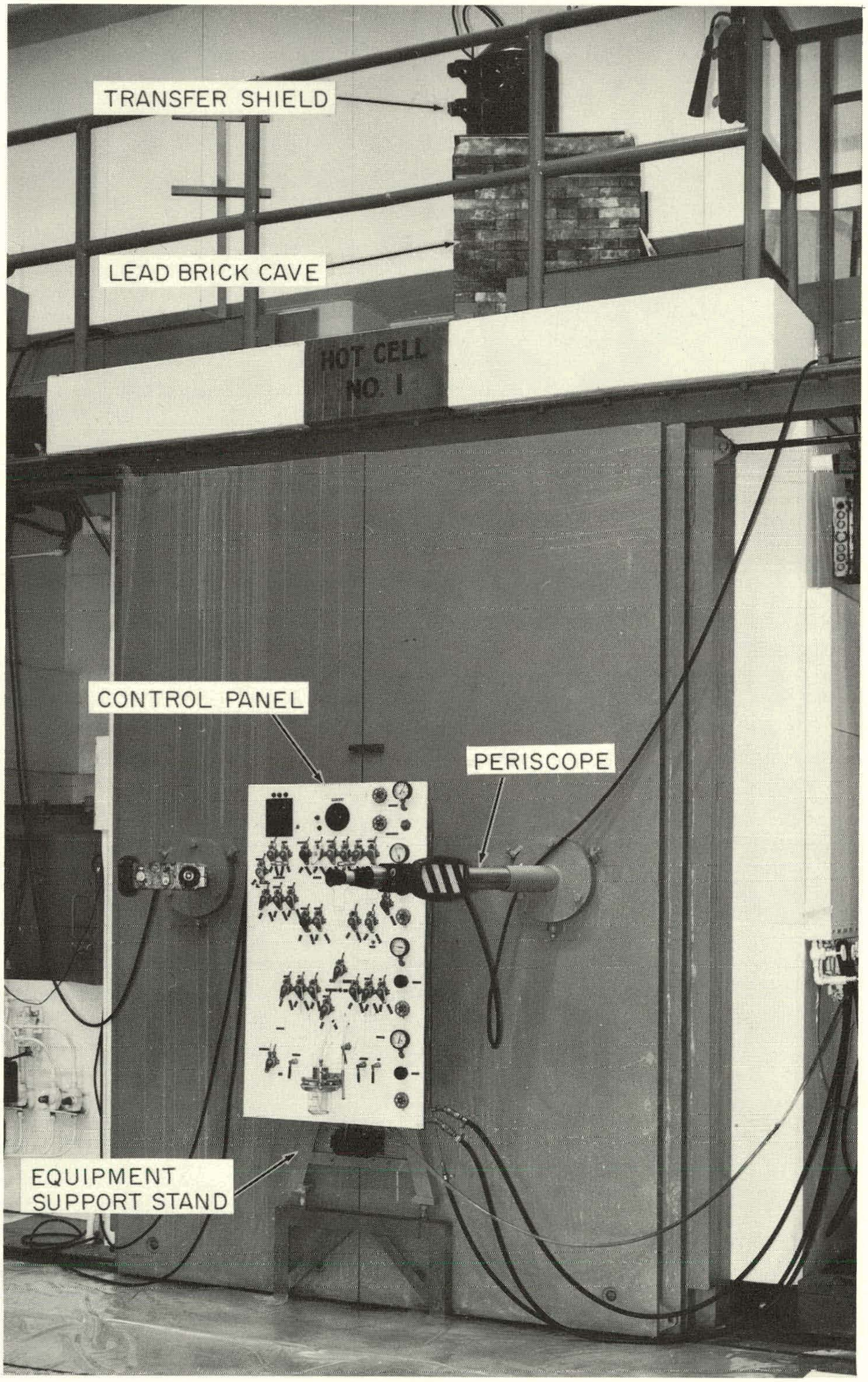

Figure 2. Hot cell for chemical processing.

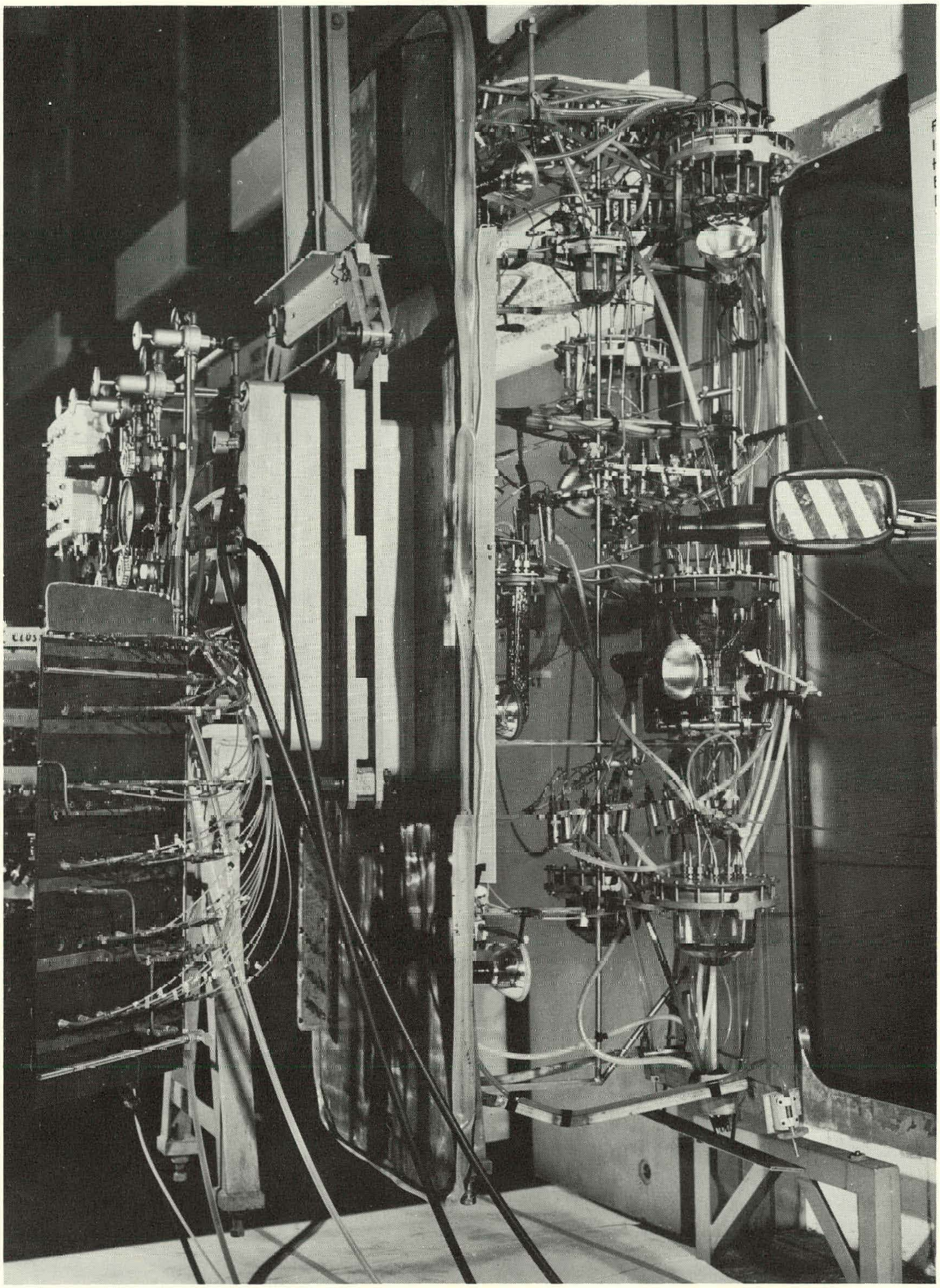

Figure 3. Remotely operated chemical processing equipment suspended in front of hot cell ready to be placed inside.

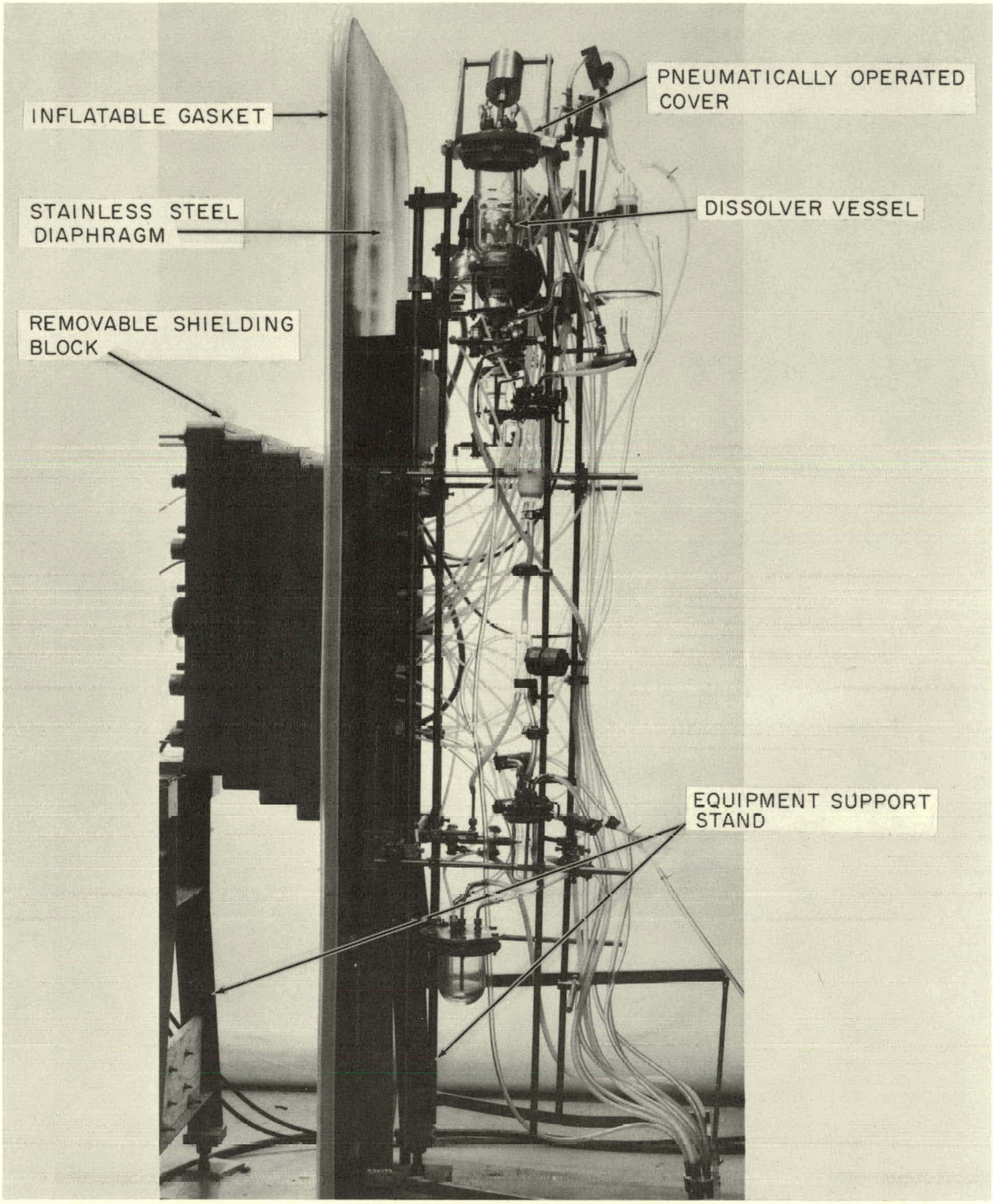

Figure 4. Remotely operated chemical processing equipment (side view) shown removed from hot cell. Removable shielding block fits into stepped opening in shielding doors. Control panel (not shown) would be at left, fastened to removable block. Stainless steel diaphragm just fits cell opening and seals cell when gasket is inflated, preventing accidental leakage of radioactive material to operating side.

Figure 5. Reaction vessel.

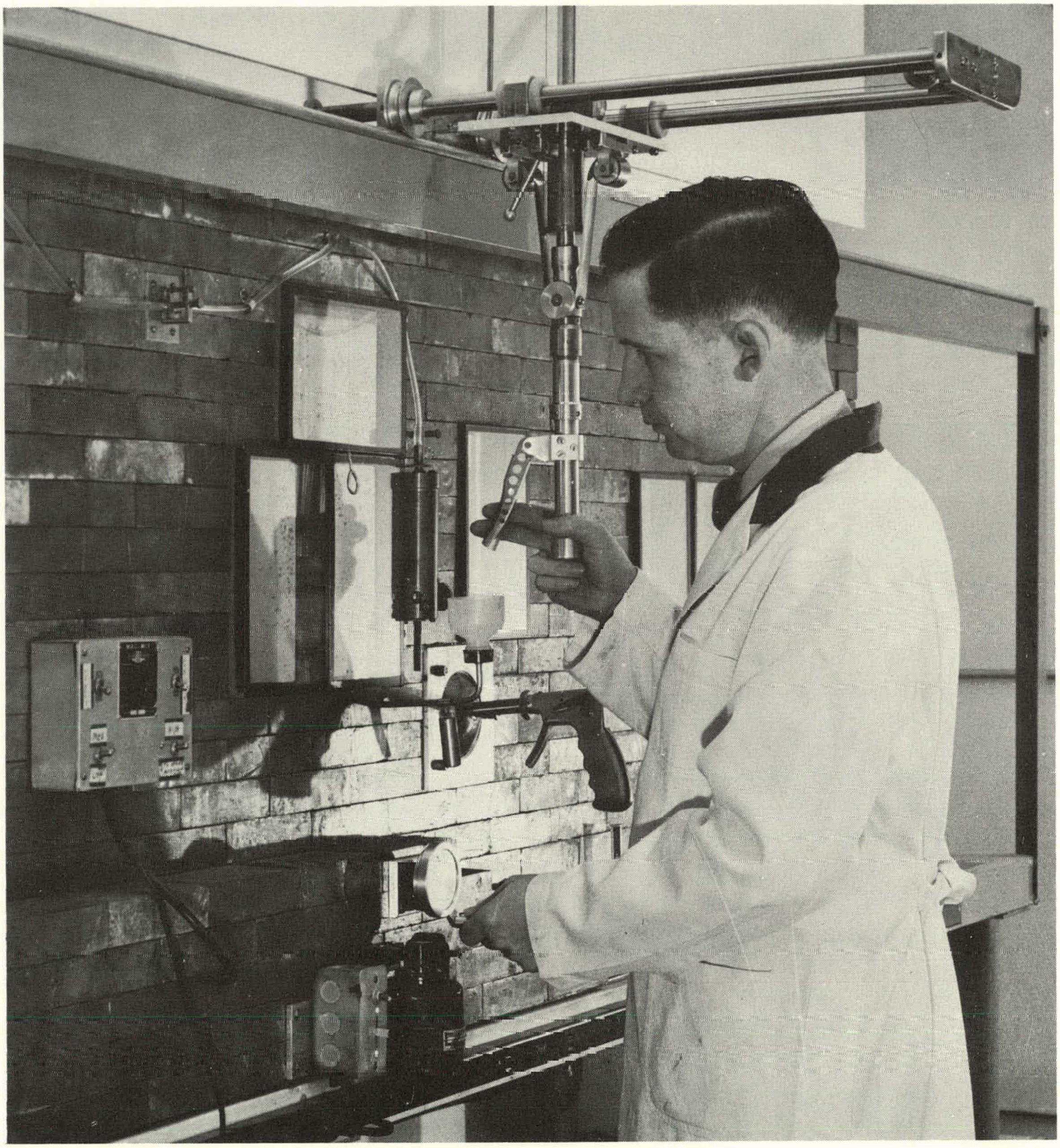

Figure 6. Semi-remote manipulation of fractional curie amounts of activity.

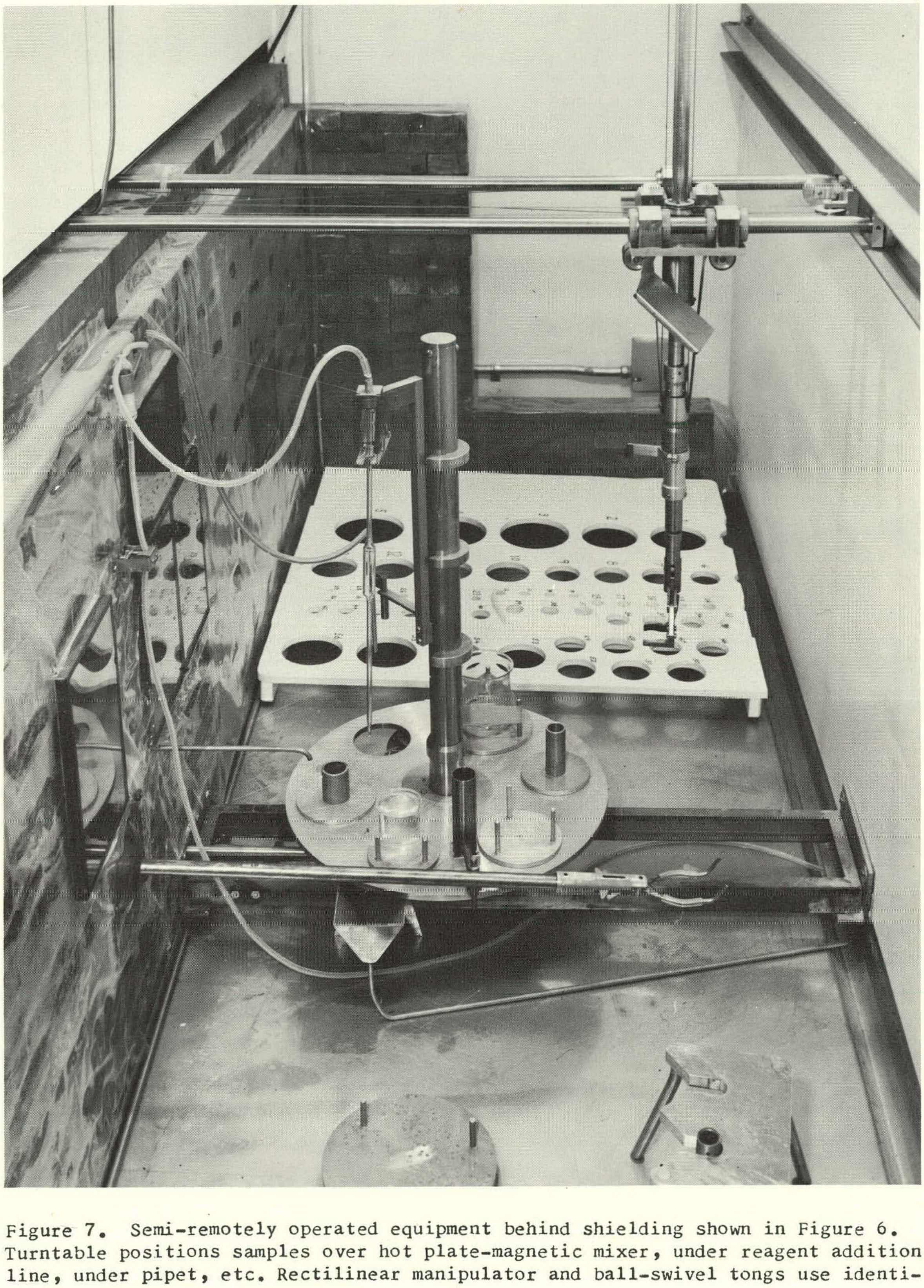

Figure 7. Semi-remotely operated equipment behind shielding shown in Figure 6. Turntable positions samples over hot plate-magnetic mixer, under reagent addition line, under pipet, etc. Rectilinear manipulator and ball-swivel tongs use identical interchangeable jaws.

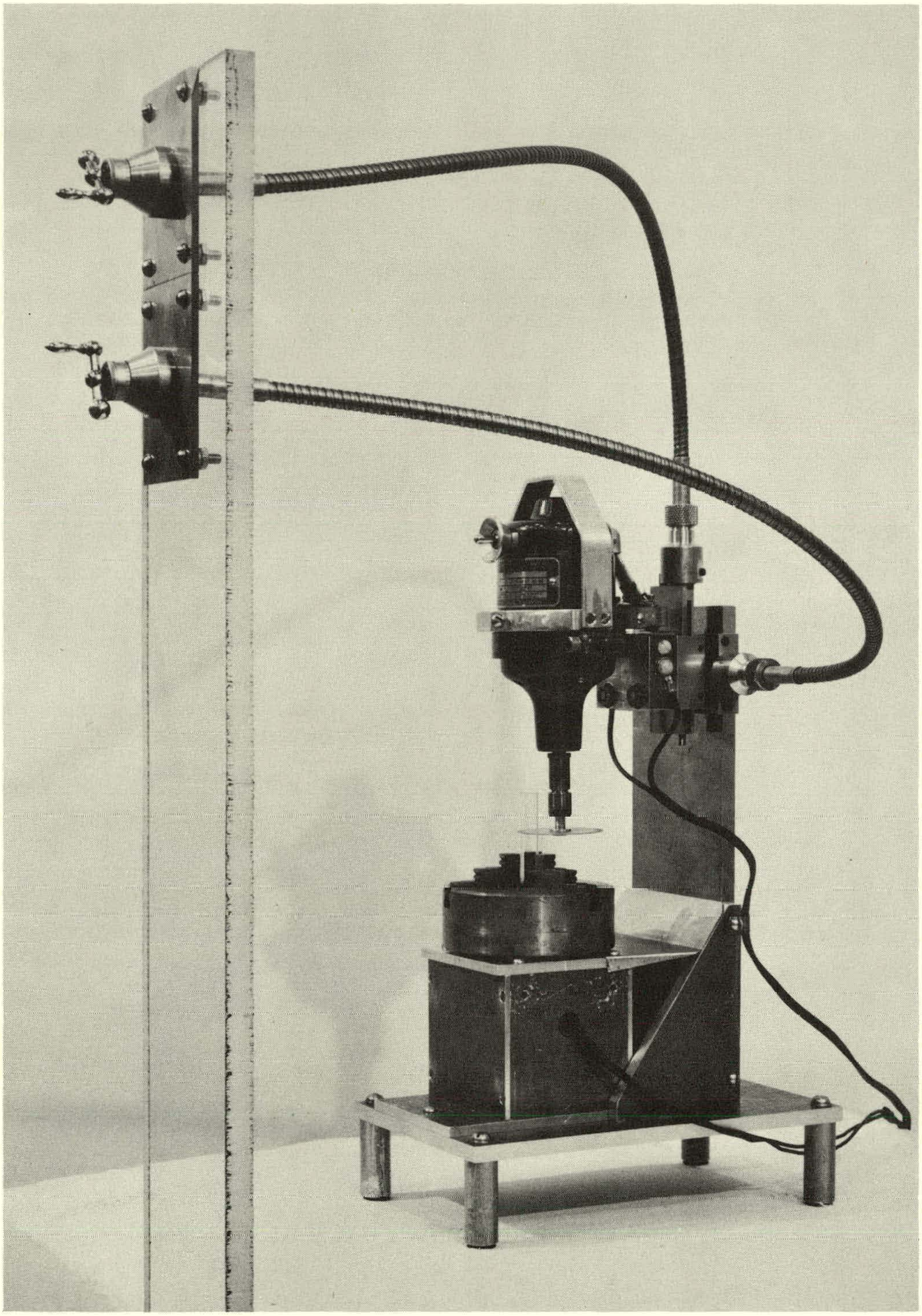

Figure 8. Remotely operated cutoff wheel for glass or quartz. Crank operated flexible shafts position wheel vertically and laterally. Vertical chuck is driven slowly by a separate motor.

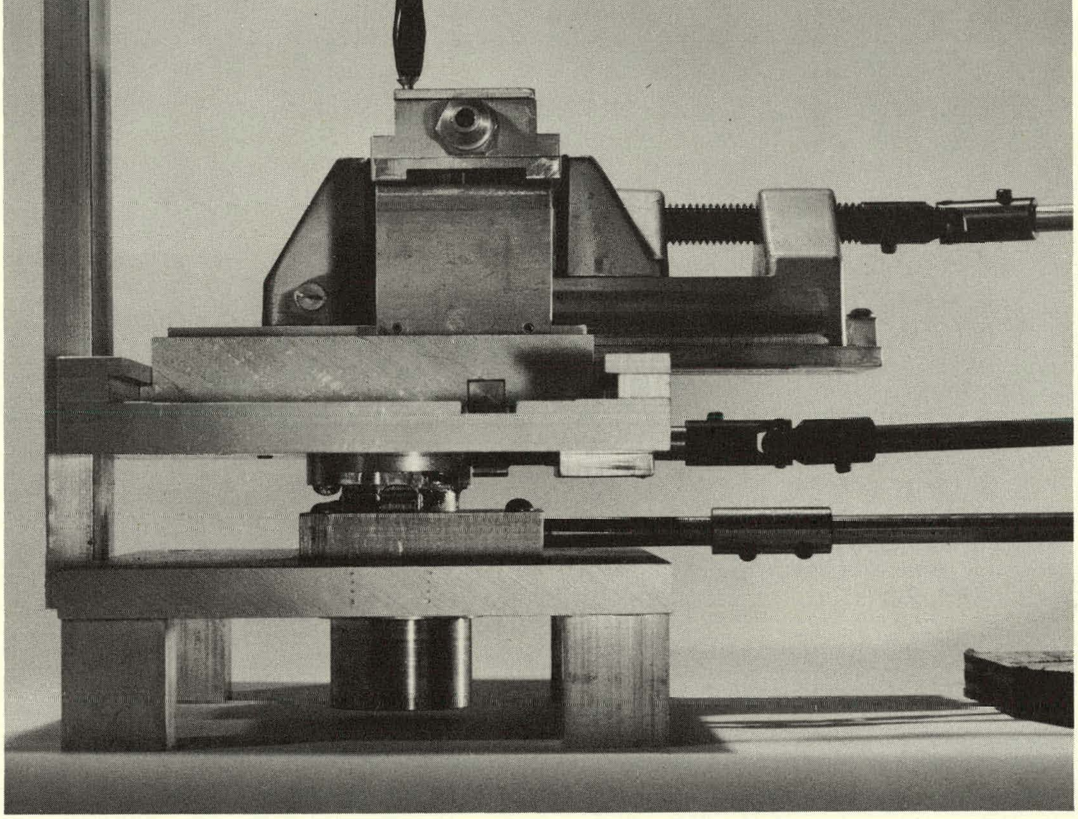

Figure 9. Remotely operated target disassembler. Rotating top shaft rotates screwdriver bit. Rotating second shaft tightens or loosens vise; pushing or pulling second shaft moves entire vise to left or right. Rotating third shaft moves vise forward and back. Rotating bottom shaft raises and lowers vise.

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Aluminum-28

Isotope: Al^{28}

Shipping Form: Generator

Parent: Mg^{28}

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

Production Method: See Magnesium-28

Generator Yield: ~30% if carrier is added; <30% without carrier

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

Product Composition: $NaAl^{28}O_2$ in 1 N NaOH

Radiochemical Purity: >99.9%

Method of Analysis: Gross decay and gamma spectroscopy

Radiations (MeV):

Beta (Al^{28})	Gamma (Al^{28})
2.87	1.78

Beta (Mg^{28})	Gamma (Mg^{28})
0.42 (100%)	0.032 (96%)
	0.40 (31%)
	0.95 (29%)
	1.35 (70%)

Special Equipment: Generator (see Figure 10)

A. Preparation of Generator

1. Over Dowex-50W (H^+ form) pass 1 - 2 M NaCl until effluent is at same pH as NaCl.	This converts the resin to the Na^+ form.
2. Wash resin with H_2O until all Cl^- is removed.	Use Ag^+ to test for presence of 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.

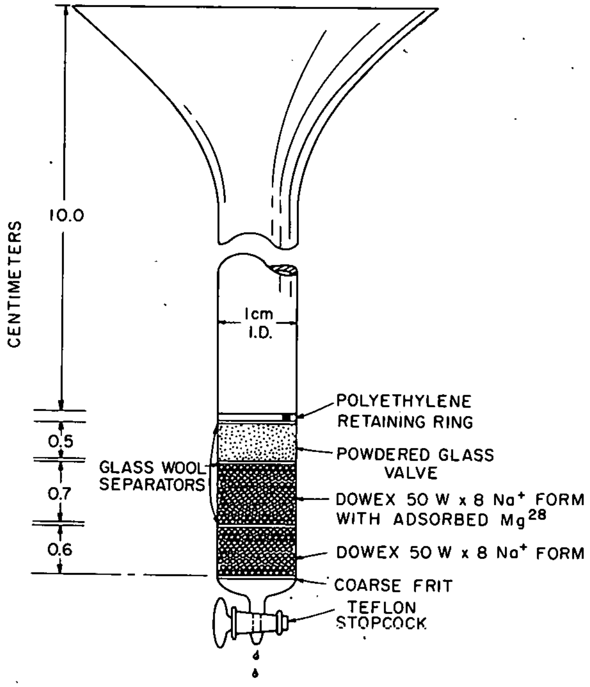

Figure 10. Aluminum-28 generator.

B. Repurification of Mg^{28}

1. Start with $Mg^{28}Cl$ solution.	This is supplied by the Hot Laboratory Division, Brookhaven National Laboratory. (See separate procedure for producing Mg^{28}.)
2. Add $\sim$5 mg each of Cu^{++} and Cd^{++} carriers.	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.
3. Dilute to 15 ml and adjust pH to 3 - 4 with 0.1 N NaOH solution.	Indicator paper is satisfactory. Too much NaOH will precipitate $Mg(OH)_2$.
4. Saturate with H_2S gas and digest on a hot plate.	Several minutes are required to form coarse particles of $Cu^{64}S$ and $Cd^{115}S$.
5. Filter through paper placed on glass frit on filter chimney.	Use Whatman #42 paper and fine frit. Save filtrate; discard precipitate.
6. To filtrate add a few drops of Brom Thymol Blue indicator.	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 H_2S.

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 Mg^{28} 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 Mg^{28} left in the supernate. (The batchwise adsorption deposits the Mg^{28} on a relatively large surface area of resin and produces higher subsequent milking yields than would be obtained if all of the Mg^{28} 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 Al^{28} with 2 - 3 ml 1 N NaOH. Alternatively, the NaOH may contain carrier Al^{3+} as desired (e. g. 0.5 mg/ml).	Milking requires 10 - 30 seconds. If carrier Al^{3+} 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:	Ar^{38}
Shipping Form:	Gas sealed in glass ampoule
Half-Life:	∞ (stable)
Production Method:	$Cl^{37}(n,\gamma)Cl^{38} \xrightarrow[\tau = 37.3\text{ m}]{\beta^-} Ar^{38}$
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} n sec^{-1} 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 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 ($\sim 10^{-7}$ 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 ~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.	Caution: A considerable amount of S^{35} from $Cl^{35}(n,p)S^{35}$ 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.
2. Evacuate the system for 24 hours.	
3. Heat the Ag getter to ~230°C and the Ti getter to ~1000°C, and bake the entire system at ~370°C for 5 - 8 hours. During the last hour play an air-gas flame over the entire system occasionally.	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^{35}. The Ti getter is on a side arm off the main train.
4. Turn off the Ti and Ag getter furnaces. Then turn off the bake oven.	This sequence prevents readsorbing in the rest of the system any condensibles remaining in the getters.
5. Measure the pressure of the system at room temperature.	Pressure should be ~10^{-7} torr.
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.	At ~650°C NaCl starts subliming into the cooler part of the system.
8. After ~1 hour refrigerate the charcoal trap with liquid N_2 for ~30 minutes.	This trap is located in the side arm between the Ti getter and the main line. This operation adsorbs Ar.
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.	

11.	After ∼90 minutes, allow the Ti getter to cool to ≤100°C.	
12.	Refrigerate the charcoal trap with liquid N_2 for ∼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.	This removes residual gaseous impurities from the system.
14.	Close this valve (step #12) and replace the liquid N_2 around the charcoal trap with a dry ice-solvent mixture.	Ar desorbs at dry ice temperature but higher boiling impurities do not.
15.	Open the valve to the sample collection section and refrigerate with liquid N_2 the charcoal trap in that section.	This transfers the Ar to the sample collection section; the volume of this section should be known in advance.
16.	When the pressure has become constant close this valve (step #14).	
17.	Remove liquid 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.	∼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:	$Ni^{64}(\alpha,p)Cu^{67}$ (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):

Beta	Gamma
0.395	0.092
0.484	0.182
0.577	0.296

Special Equipment: Electrolysis apparatus.

A. Target Preparation and Irradiation

1. Silver solder gold foil onto a copper target blank.	Blank must be capable of being water-cooled during irradiation. Good contact between gold and copper is required for heat transfer.
2. Electroplate enriched Ni^{64} from ammoniacal solution onto gold foil, with stirring.	$(NO_3)^-$ 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.	Use torch to heat copper blank to bright red.
2. Place foil in 50 ml beaker, nickel side down on a few drops of hot 8 N HNO_3.	This position minimizes dissolution of the solder.
3. After Ni has dissolved, rinse foil with H_2O, add 3 drops conc. H_2SO_4, and transfer quantitatively to a special electrolysis vessel clamped in a warm (60°C.) water bath.	Vessel may have side arms for electrodes and should have a cover to prevent loss from spattering due to gas evolution.
4. Insert platinum wire electrodes.	
5. Add ~0.5 mg each of Co^{++}, In^{+++}, Cd^{++}, and Cu^{++}.	Co, In, and Cd activities leach out of Au foil and will coprecipitate with Cu unless these holdback carriers are added.
6. Apply 2.4 - 2.6 V DC to electrodes for ~45 m.	Cu^{67} and added Cu will plate out on cathode.
7. Without reducing applied voltage remove cathode and promptly rinse with H_2O.	If voltage is shut off prior to cathode removal, Cu will redissolve.
8. Dissolve product by running 1 - 2 drops 6 M HNO_3 down the wire.	

C. Reclamation of Ni^{64}

1. Make Ni solution basic with an excess of conc. NH_4OH.	
2. Filter and discard any precipitate.	This removes ions which interfere with subsequent preparation of electrolysis solution.
3. To filtrate add solution of 0.01 N dimethylglyoxime.	This precipitates nickel dimethylglyoxime.
4. Digest near boiling.	This permits particle growth and easier subsequent filtration.

5. Cool, filter, and wash, discarding filtrate.	Filter through paper.
6. Place the filter paper and the precipitate in a beaker and add 40 ml conc. HNO_3 and 15 ml conc. $HClO_4$.	This will completely oxidize and dissolve both precipitate and paper, volatilizing organic components.
7. Evaporate to dryness.	
8. Dissolve residue in a minimum of 9 <u>N</u> H_2SO_4 and neutralize with concentrated NH_4OH.	Solution is ready for electroplating a new target.

Fluorine-18

Isotope:	F^{18}	
Half-Life:	110 m	
Production Method:	$Li^{6}(n,t)He^{4}$; $O^{16}(t,n)F^{18}$ (reactor)	
Cross Section:	950 b for $Li^{6}(n,t)He^{4}$	
Target:	Li_2CO_3 (unenriched)	
Target Weight:	10 g	
Neutron Flux:	1.2×10^{13} n sec^{-1} cm^{-2}	
Irradiation Time:	~6 h	
Reactor Yield:	~40 mC	
Chemical Yield:	70 - 80%	
Specific Activity:	"Carrier-free" F^{18} (i. e. no F carrier added)	
Product Composition:	NaF^{18} + trace of $NaAlO_2$ in 0.1 $\underline{M}$ NaOH	
Radiochemical Purity:	>99.99% F^{18}; trace of H^3	
Method of Analysis:	Well-type scintillation counter standardized against 4π β counter	
Radiations (MeV):	Positron	Gamma
	0.65 (100%)	0.51 (annihilation gammas)
Special Equipment:	Glove box or fume hood suitable for handling H^3 contamination. Isotope can opening tools and crimping press for closing. 1½-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 Li_2CO_3.	Annular space should be $\sim$1/8 inch thick. Because of the high σ of 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 Li_2CO_3 powder to Erlenmeyer flask containing $\sim$20 ml H_2O and magnetic stirring bar.	Use wide neck funnel and rinse with H_2O.
2. Add conc. HCl slowly with stirring to dissolve Li_2CO_3.	Avoid rapid CO_2 evolution.
3. Adjust pH to acid side of Brom Thymol Blue indicator with NaOH solution.	
4. Pass solution through alumina column, using vacuum to attain 1 - 2 ml/min.	Column preparation: Slurry 10 g Woelm alumina (neutral grade) with 50 ml 0.5 $\underline{M}$ HCl, decanting and discarding fines. Wash with two 50-ml portions of H_2O, 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 H_2O. A pad of glass wool on top of the alumina will prevent it from plugging.
5. Wash column with 30 ml H_2O containing 1 drop conc. HCl.	Use vacuum if necessary.
6. Elute F^{18} with 40 ml 0.1 $\underline{N}$ NaOH containing Brom Thymol Blue indicator. Collect in 3 portions:	Collect portions in large test tubes inside side-arm flask on which slight vacuum is pulled.

#1 - until blue color starts to come through frit,	10 - 12 ml.
#2 - next 15 ml,	Contains most of the F^{18}.
#3 - remainder of effluent.	
7. Assay portion #2 and transfer to shipping bottle.	Count annihilation gammas in calibrated well-type scintillation counter.

Note: Product is an aluminum fluoride complex, which may be converted to NaF by distillation from conc. H_2SO_4, collecting distillate in dilute NaOH.

Gallium-68

Isotope:	Ga^{68}
Shipping Form:	Generator
Parent:	Ge^{68}
Half-Lives:	Ga^{68}, 68 m; Ge^{68}, 275 d
Production Method:	$Ga^{69}(p,2n)Ge^{68}$ (ORNL cyclotron; see Isotope Sales Department, ORNL, for details)
Generator Yield:	~70%
Specific Activity:	Carrier-free
Product Composition:	Ga^{68}-EDTA complex in 0.005 M EDTA; or alternatively as $Ga^{68}Cl_3$ in ether; or alternatively as $(Ga^{68})_2(SO_4)_3$ "weightless" solid.
Radiochemical Purity:	99.999% Ga^{68}; 0.001% Ge^{68}
Method of Analysis:	Gross decay
Radiations (MeV):	

Positron (Ga^{68})	Gamma (Ga^{68})
0.82 (1.5%)	0.51 (annihilation gammas)
1.894 (86 %)	1.08 (~4%)
Particle (Ge^{68})	Gamma (Ge^{68})
none	only characteristic Ga^{68} X rays; (100% electron capture)

A. Preparation of Generator

1. Wash 10 g alumina with H_2O 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.	
3. Adjust pH to 7.	Solution as received commercially is strongly acid.
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 Ga^{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 Ga^{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: I^{124}

Half-Life: 4.2 d

Production Method: $\mathrm{Sb}^{121}(\alpha,n)\mathrm{I}^{124}$ (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 I^{124} after 48 h cooling

Chemical Yield: ~80%

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

Product Composition: HI^{124} in $\mathrm{NaHSO_3}$ solution or in $\mathrm{Na_2SO_3}$ solution

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

Method of Analysis: Gamma spectroscopy and gross decay

Radiations (MeV):

Positron	Gamma
~0.90 (0.2%)	0.603 to 2.745 } >19 γ's
1.55 (14 %)	
2.15 (11 %)	

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.	Holder is a flat metal plate with a recess to fit target and a bolted-on retaining frame.
4. Cover target with aluminum foil; bolt 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.	Use a manipulator-held scribing point.
2. Remove and discard foil and dump target into still pot containing chromic acid-sulfuric acid-iodide carrier mixture.	Use tweezers or suitable manipulator. Mixture preparation: Dissolve 13 g CrO_3 in 70 ml H_2O; add 280 ml conc. H_2SO_4 and 5 μg I^- carrier.
3. After target dissolution cool solution and add 100 ml H_2O and 10 ml 30% H_3PO_3.	This reduces the iodates to a distillable form.
4. Heat and distill into H_2SO_3 solution, adding 1 - 2 drops 30% H_2O_2 occasionally.	H_2O_2 releases O_2 gas which acts as a carrier. H_2SO_3 traps radioiodines in non-volatile form. Stop distillation when activity ceases to distill.
5. To distillate add 1 g oxalic acid and 18 - 20 ml conc. H_2SO_4 and redistill into H_2SO_3 solution.	This removes any Sb carried over during the first distillation. No H_2O_2 is needed here because of small volumes.
6. If desired, the product may be further concentrated by making basic and evaporating down.	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 H_2SO_3.

Iodine-132

Isotope:	I^{132}
Shipping Form:	Generator
Parent:	Te^{132}
Half-Lives:	I^{132}, 2.3 h; Te^{132}, 77 h
Production Method:	See Tellurium-132
Generator Yield:	~70%
Specific Activity:	Carrier-free
Product Composition:	NaI^{132} in 0.01 M NH_4OH; traces of stable Al and Na
Radiochemical Purity:	$<10^{-3}$% Te^{132}; <0.01% - 0.3% I^{131}, depending on history of generator (see note below)
Method of Analysis:	Well-type scintillation counter standardized against 4π β counter

Radiations (MeV):

Beta (I^{132})	Gamma (I^{132})
0.80 (21 %)	0.528
1.04 (15 %)	0.670
1.22 (12 %)	0.777
1.49 (12 %)	0.96
1.61 (21 %)	and >23 other γ's from 0.240 to 2.9
2.14 (18 %)	
2.92 (0.03%)	

Beta (Te^{132})	Gamma (Te^{132})
0.22	0.052
	0.233

Special Equipment: Remotely operated dispensing equipment.

A. Preparation of Generator

1. Load a glass Büchner 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_2O until effluent is clear.	
4. Pass 50 ml 1 M HNO_3 through the alumina.	

B. Loading of Generator

1. Into a few ml H_2O pipet the required amount of Te^{132} product solution and make up volume to 25 ml with H_2O.	Use solution from step #25 of Te^{132} process.
2. Mix well.	Remotely operated magnetic stirrer is convenient.
3. Drip into generator (see above) at <1 drop/sec.	
4. Pass 30 ml H_2O through generator.	
5. Pass 10 ml 1 M NH_4OH through generator.	This neutralizes any residual free acid left on the alumina.
6. Pass 500 ml 0.01 M NH_4OH through generator.	

Note: Generator is milked (i. e. I^{132} is eluted from alumina) with 10 - 20 ml 0.01 M NH_4OH. Washing the resin with 0.01 M NH_4OH prior to a given milking decreases the I^{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 I^{131} contamination also decreases with increasing age of generator and with decreasing "growth time" (defined as the time between a given milking and the previous milking or wash). Thus, for minimum I^{131} contamination one would choose an old generator and wash it with 300 ml 0.01 M NH_4OH at whatever time prior to milking would allow only the desired amount of I^{132} to grow in (i. e. at that time which provides the minimum growth time for the I^{132} desired).

Iodine-133

Isotope: I^{133}

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

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

Fission Cross Section: 582 b

Target: U_3O_8 (unenriched)

Target Weight: 2 g

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

Irradiation Time: 20 h

Fission Yield: $\sim 6.9\%$

Reactor Yield: }
Chemical Yield: } This process produces ~ 100 mC I^{133}.

Specific Activity: "Carrier-free" I^{133} (i. e. no I carrier added)

Product Composition: $NaI^* + NaI^*O_3$ in dilute NaOH

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

Method of Analysis: 4π β count to determine total activity at reference time; gross decay curve on previous batches to determine relative proportions of various radioiodines.

Radiations (MeV):

Beta (I^{133})	Gamma (I^{133})
0.89	0.233
1.22	0.530
1.54	0.87

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 U_3O_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 U_3O_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 H_2SO_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 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 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.	The first 4 ml usually contains only little activity.
6. When the aluminum foil wrapping is completely dissolved add 30 ml H_2O slowly.	
7. Add 25 ml 30% H_2O_2 2 - 3 drops per second.	The 4 ml collected during addition of the H_2O_2 contains the bulk of the activity.
8. Turn off heat when 12 - 16 ml product have been collected.	
9. Select most radioactive fraction for assay and shipping.	

Magnesium-28

Isotope: Mg^{28}

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

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

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

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

Target Weight: ~12 mg

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

Irradiation Time: ~3 days

Reactor Yield: ~100 μC Mg^{28}-Al^{28}

Chemical Yield: ~90%

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

Product Composition: $Mg^{28}Cl_2$ in dilute HCl containing <1% NaCl

Radiochemical Purity: 95% Mg^{28}-Al^{28}, 5% H^{3}, traces of Cu^{64}, Cd^{115} as of 24 hours following processing

Method of Analysis: Well-type scintillation counter standardized against 4π β counter

Radiations (MeV):

Beta (Mg^{28})	Gamma (Mg^{28})
0.42 (100%)	0.032 (96%)
	0.40 (31%)
	0.95 (29%)
	1.35 (70%)

Beta (Al^{28})	Gamma (Al^{28})
2.87	1.78

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^{26} metals separately and rapidly on an analytical balance.	Use Li^6:$Mg^{26} \approx 3:1$ by weight. Use Li^6 enriched to ~96% and Mg^{26} enriched to ~99%. Prevent air oxidation.
2.	Rapidly transfer metal samples into dry box air lock and flush with dry argon.	Ordinary tank argon is sufficiently dry for this purpose.
3.	Transfer metal samples from air lock into dry box proper.	Dry box should already have been filled with argon. Argon flow rate need only be sufficient to prevent back diffusion of air into box.
4.	Melt samples together in graphite crucible at ~650°F in furnace, stirring with a graphite rod.	As soon as all is molten, proceed to next step.
5.	Pour molten alloy into shallow graphite mold.	Rapid cooling will insure homogeneity of alloy.
6.	Place ingot under oil for transfer out of box.	Prevent air oxidation. Mineral oil is satisfactory for this purpose.
7.	Using a suitable small rolling mill, roll to 0.25 mm thickness.	If target is too thick, the high σ of Li^6 will seriously depress the internal neutron flux, reducing the specific activity of Mg^{28}.
8.	Cut into suitable strips for irradiation.	Cuts easily with ordinary scissors.
9.	Store under oil	Prevent air oxidation.

B. Target Irradiation

1.	Clean alloy foil with ethyl acetate, weigh, and insert in slits in 3/4-inch diameter graphite rod.	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.
2.	Place loaded rod in suitable aluminum can and weld shut.	Cap of can should have short length of 1/8-inch diameter tube attached to it.
3.	Evacuate can via tube on cap and fill with helium.	Ordinary oil-pumped helium is satisfactory. Helium prevents air oxidation and is a good heat transfer agent.

4.	Crimp tube closed.	End need not be welded if crimp seal is a good one.
5.	Irradiate in reactor ~3 days.	Use cooled hole to prevent sample from melting. Note: If sample melts, thickness will increase and specific activity of Mg^{28} will decrease; see above.
6.	Open can with remotely operated opener.	Enclosure must be capable of dealing with considerable tritium contamination from $Li^6(n,\alpha)H^3$.

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^{18}, H^3 and Na^{24}. F^{18} comes from (t,n) reaction on oxide impurities. H^3 comes from unreacted tritons. Na^{24} comes from (n,γ) reaction on sodium impurities, from (t,α) reaction on residual Mg^{25}, and from (n,α) reaction on Al impurities.
5.	Wash twice with ice water containing 3 drops of 3 N NaOH, discarding supernate.	Reduces H^3 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.	
9.	Add 0.1 N NaOH until just yellow.	This precipitates $Al(OH)_3$. The end point is critical. $Mg(OH)_2$ will precipitate if pH goes too high.

10. Centrifuge and wash precipitate with H_2O.	Combine supernate and washes and save. Discard precipitate.
11. To supernate add 3 mg F^- carrier and 1 drop of 3 N HCl.	Use KF for carrier, 30 mg F^- per ml.
12. Pour supernate onto prepared ion exchange column.	Column preparation: In a 1-inch fritted Büchner funnel settle 3/4 inch of Dowex 1 x 8 (Cl^- form). Cover with cloth disc and add 3/4 inch of Woelm Al_2O_3 (neutral grade) which had been previously conditioned with 0.5 N HCl and washed twice with H_2O. Column removes remainder of F^{18}. This is necessary for subsequent Mg^{28} assay.
13. Wash column with 75 ml of 0.001 N HCl.	
14. Combine effluents and transfer to distillation flask. Distill to near dryness.	Use glass beads in flask to prevent bumping. Discard distillate. Steps #14 - #16 reduce volume of product (residual solution) and H^3 contamination in it.
15. Add ~50 ml 0.001 N HCl and distill to near dryness.	
16. Repeat step #15.	
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^{99}

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

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

Fission Cross Section: 582 b

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

Target Weight: ~180 mg U^{235}; ~7 g Al

Neutron Flux: ~1.2 x 10^{13} n sec^{-1} cm^{-2}

Irradiation Time: ~3 d

Fission Yield: ~6%

Reactor Yield: ~2.3 C Mo^{99}

Chemical Yield: ~70%

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

Product Composition: $(NH_4)_2Mo^{99}O_4$ in 1 M NH_4OH

Radiochemical Purity: >99.99% Mo^{99}, traces of I^{131} and Ru^{103} (at time of shipment)

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

Radiations (MeV):

Beta (Mo^{99})	Gamma (Mo^{99})
0.45 (14%)	0.041
0.87 (~1%)	0.181
1.23 (85%)	0.372
	0.740
	0.780
	0.950

Beta (Tc^{99m})	Gamma (Tc^{99m})
<0.03% primary β^-	0.002
9.5% internal conversion electrons	0.140
	0.142

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 ~180 mg U^{235}, trimming off excess aluminum at edges.	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 ~1/4 inch beyond the alloy.
2. Place alloy in suitable aluminum can and weld shut.	Cap of can should have short length of 1/8-inch-diameter tube attached to it.
3. Evacuate can via tube on cap and fill with helium.	Helium prevents air oxidation and is a good heat transfer agent.
4. Crimp tube closed.	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.	Use Fisher chromatographic alumina, 50 - 200 mesh.
2. Slurry the alumina into the column.	See schematic diagram of equipment (Figure 11).
3. Slurry onto the alumina a layer of ground glass.	This keeps subsequently added reagents from disturbing the alumina bed.
4. Wash the alumina with the following in this order: 200 ml 1 $\underline{M}$ HNO_3, 100 ml H_2O, 100 ml 1 $\underline{M}$ NH_4OH, 200 ml H_2O, 100 ml 1 $\underline{M}$ HNO_3	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.	See introductory section preceding these individual procedures for a description of this equipment and its operation.
2. Open can remotely and drop irradiated target into the dissolver vessel in the cell.	

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.	This prevents any subsequently added liquid from draining through the frit.
2. Cover the irradiated target with 48 ml 6 $\underline{M}$ HNO_3 and 2 ml 2 $\underline{M}$ $Hg(NO_3)_2$.	If this volume fails to cover the target completely add enough 6 $\underline{M}$ HNO_3 to do so. The $Hg(NO_3)_2$ catalyzes the dissolution.
3. Apply heat to start the dissolution. Turn off heat when the reaction becomes vigorous.	Caution! Reaction can become violent.
4. Cautiously add 3 more 50-ml portions of 6 $\underline{M}$ HNO_3, pausing between additions.	Caution! Reaction will run away if acid is added too rapidly.
5. After solution is clear, add 5 ml Te carrier solution (0.1 mg Te/ml 5 $\underline{M}$ HNO_3).	It may be necessary to wait a few minutes for solution to clear before adding Te; it may also be necessary to add an additional 50-ml portion of 6 $\underline{M}$ HNO_3 to clear the solution.
6. Wash Te carrier into dissolver with a few ml 6 $\underline{M}$ HNO_3.	This avoids loss of carrier by hold-up in inlet lines.
7. Add H_2O 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.	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 HNO_3 and filter onto alumina column.	
10. Run 2 200-ml portions of 1 M HNO_3 onto alumina column.	This washes unadsorbed ions off the alumina.
11. Run 100 ml H_2O onto alumina column, followed by 100 ml 0.01 M NH_4OH.	This removes any traces of acid and makes the alumina slightly basic.
12. Run 145 ml 1 M NH_4OH 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 NH_4OH elutes Mo^{99}, but none appears in the first 20 ml.

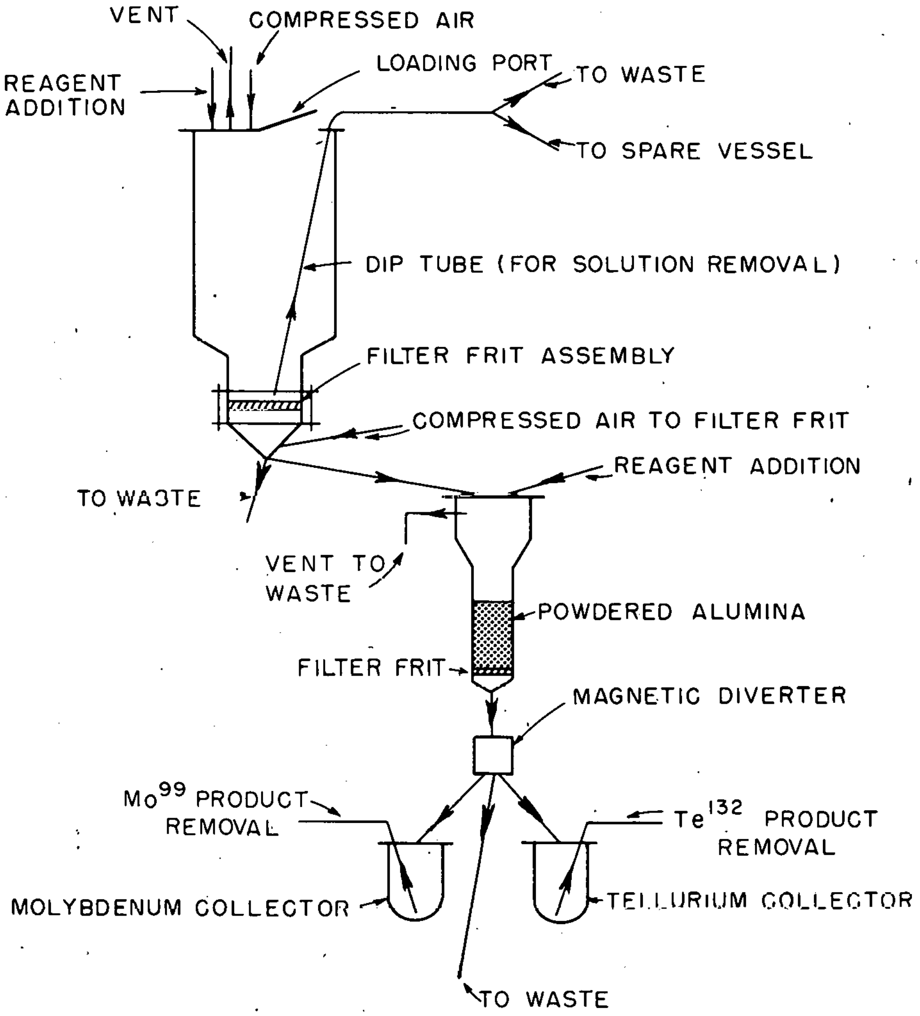

Figure 11. Fission product separation equipment.

Potassium-43

Isotope: K^{43}

Half-Life: 22 h

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

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

Specific Activity: Carrier-free

Product Composition: K^{43} in H_2O, saline solution, etc. as required

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

Method of Analysis: Precalibrated well-type scintillation crystal

Radiations (MeV):

Beta	Gamma
0.47 (8 %)	0.22 (3%)
0.83 (87 %)	0.37 (85%)
1.24 (3.5%)	0.39 (18%)
1.81 (1.5%)	0.59 (13%)
	0.61 (81%)
	1.01 (2%)

Special Equipment: Specially designed container for irradiating gas. Tongs and fixtures for semi-remote handling of irradiated target. Hood for disposing of radioactive argon. Vacuum pump.

A. Target Preparation

1. Evacuate target chamber (to 10 - 20 μ) and fill with argon.	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 α energy to minimize competing $Ar^{40}(\alpha,pn)K^{43}$ reaction.
2. Repeat step #1 several times.	
3. Fill with argon to 15 psig.	

B. Post-Irradiation Processing

1. Open the top valve on the target chamber, releasing the argon in a hood.	Use a vise to hold the chamber upright.
2. Through a funnel inserted into the upper valve, add 10 ml suitable rinse solution.	Rinse may be pure H_2O, physiological saline, or any aqueous solution as required.
3. Close both valves. Shake and rotate chamber so that all inner surfaces are wet.	Atomic K^{43} is on inside walls and readily dissolves.
4. Drain solution through bottom valve into collection bottle.	
5. Repeat steps #1 - #4.	

<u>Note</u>: Rinse target chamber out thoroughly to prevent corrosion prior to re-use.

Scandium-48

Isotope:	Sc^{48}
Half-Life:	44 h
Production Method:	$V^{51}(n,\alpha)Sc^{48}$ (Cockroft-Walton generators)
Production Cross Section:	18 mb
Target:	V_2O_5
Target Weight:	100 g
Neutron Flux:	$\sim 5 \times 10^8$ n sec^{-1} cm^{-2}
Neutron Source:	$H^3(d,n)He^4$
Beam Energy:	Fast neutrons are required; 14 - 15 MeV neutrons are produced from ~150 keV deuterons.
Integrated Beam Current:	~30 μAh of deuterons
Accelerator Yield:	~2 μC
Chemical Yield:	50%
Specific Activity:	Carrier-free
Product Composition:	$Sc^{48}Cl_3$ ("weightless" solid)
Radiochemical Purity:	>99.3%
Method of Analysis:	Gamma spectroscopy and decay of 175 keV γ

Radiations (MeV):	Beta	Gamma
	0.65 (93%)	0.175 (7%)
	0.48 (7%)	0.98 (100%)
		1.04 (100%)
		1.31 (100%)

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 H^3 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.	
3. With stirring pour slowly into 2000 ml 2 <u>M</u> HCl.	Separation of a red solid should be avoided although a small amount is not troublesome.
4. Add 10 ml 1 <u>M</u> $FeCl_3$.	
5. With stirring add slowly 1000 ml 4 <u>M</u> NaOH.	$Fe(OH)_3$ precipitates, carrying Sc^{48} and some V.
6. Centrifuge, discarding supernate.	
7. Dissolve precipitate in HCl and reprecipitate with NaOH.	Concentrations are unimportant, but volumes should be kept small. This step removes the remaining V.
8. Repeat steps #6 - #7 twice.	
9. Dissolve precipitate in 100 ml 8 <u>M</u> HCl.	
10. Extract with 100 ml diethyl ether.	Use ether which has been pre-equilibrated with 8 <u>M</u> HCl.
11. Partially neutralize aqueous layer with conc. NH_4OH.	The solution gets hot, the residual ether boils off, and some Sc^{48} is lost.
12. Add 11 mg Fe^{3+} carrier and reprecipitate with NH_4OH.	
13. Repeat steps #6 - #7 several times, substituting NH_4OH for NaOH.	If any tetravalent V is present, a blue precipitate, insoluble in acid, will appear. To remove it, centrifuge the solution while acid. No Sc^{48} is lost on this precipitate.

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 H_2O, discarding washes.	This removes salts (viz. NH_4Cl) 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 Sc^{48} into tri-n-butyl phosphate, and back extracting with H_2O 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 µAh	
Cyclotron Yield:	>100 µC Y^{87}/µAh	
Chemical Yield:	>90% Y^{87}	
Generator Yield:	~70% Sr^{87m}	
Specific Activity:	"Carrier-free" Sr^{87m} (i. e. no Sr carrier added)	
Product Composition:	$(Sr^{87m})_3(C_8H_5O_7)_2$ 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):	Beta (Sr^{87m})	Gamma (Sr^{87m})
	none (~25% conversion electrons)	0.39
	Beta (Y^{87})	Gamma (Y^{87})
	0.3% β^+	0.483
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.
2. Melt the RbCl (m.p. ∼715°C) under an inert atmosphere. Cool.	Prevent oxidation of copper.
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.	
2. Add 25 ml Dowex 1 x 8 resin (50 - 100 mesh).	Do not disturb quartz.
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 H_2O until no Cl^- is detectable.	Requires 100 - 200 ml H_2O. Use $AgNO_3$ solution to detect Cl^-.
5. Pass 200 ml 0.5 N $NaHCO_3$ solution through column, followed by 200 ml H_2O.	This puts column in HCO_3^- form.

C. Post-Irradiation Processing of Parent Y^{87}

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.	Use coarse Büchner funnel to remove particles of copper corrosion.
4. Cover copper target strip with diluted Y carrier solution.	Diluted carrier solution: 17 ml H_2O + 1 drop 6 N HCl + 1 ml Y carrier (3.5 mg Y/ml).
5. Filter solution into centrifuge tube, rinsing Lucite vessel with H_2O and filtering rinse into centrifuge tube.	
6. Remove copper strip from work area.	This will reduce radiation level.
7. Add 20 drops conc. NH_4OH to solution in centrifuge with swirling.	$Y(OH)_3$ precipitates.
8. Centrifuge, discarding supernate.	~5 min is sufficient.
9. Dissolve precipitate with 6 drops 6 N HCl.	
10. Reprecipitate with 6 drops conc. NH_4OH.	
11. Repeat step #8.	
12. Add 1 ml H_2O to precipitate; then dissolve by adding 3 - 6 drops 3 N HCl.	Solution should be clear.
13. Add ~10 ml H_2O and 10 ml 0.5% citric acid solution. Mix.	pH of citric acid solution should be 5.5.
14. Adjust pH to 2 - 2.5, using NaOH solution.	
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 1 - 2 ml/min flow rate. Assay an aliquot to determine amount of solution to be thus loaded.

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^{87m} 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:	$(Tc^{99m}O_4)^-$ in 0.1 M HCl	
Radiochemical Purity:	>99.99% Tc^{99m} at time of milking; traces of I^{131} and Ru^{103}	
Method of Analysis:	Well-type scintillation counter standardized against γ spectrometer	
Radiations (MeV):	Beta (Tc^{99m})	Gamma (Tc^{99m})
	<0.03% primary β^-	0.002
	9.5% internal conversion electrons	0.140
		0.142
	Beta (Mo^{99})	Gamma (Mo^{99})
	0.45 (14%)	0.041
	0.87 (~1%)	0.181
	1.23 (85%)	0.372
		0.740
		0.780
		0.950
Special Equipment:	Remotely operated dispensing equipment. ½-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 Büchner funnel with the alumina.	Use a 25 mm dia. x 85 mm high funnel having a coarse glass frit.
3. Cover with Feon cloth, held down by a polyethylene retainer ring.	"Feon" is a non-absorbing acrylic fiber filter cloth.
4. Wash with 25 ml 0.1 M HNO_3.	

B. Loading the Generator

1. Into a few ml H_2O pipet the required amount of Mo^{99} product solution.	Use solution from step #D12 of Mo^{99} process.
2. Add several drops Crystal Violet indicator solution.	
3. With stirring, add HNO_3 to indicator endpoint (pH = 1 - 2).	Remotely operated magnetic stirrer is convenient.
4. With stirring, add H_2O 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. Tc^{99m} 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: $U^{235}(n,f)Te^{132}$ + ... (reactor)

Fission Cross Section: 582 b

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

Target Weight: ~180 mg U^{235}; ~7 g Al

Neutron Flux: ~1.2 x 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 $\underline{M}$ 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 4π β counter

Radiations (MeV):

Beta (Te^{132})	Gamma (Te^{132})
0.22	0.052
	0.233

Beta (I^{132})	Gamma (I^{132})
0.80 (21 %)	0.528
1.04 (15 %)	0.670
1.22 (12 %)	0.777
1.49 (12 %)	0.96
1.61 (21 %)	and >23 other γ's from 0.240 to 2.9 MeV
2.14 (18 %)	
2.92 (0.03%)	

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.

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

D. Post-Irradiation Processing (continued)

13.	Run 400 ml conc. NH_4OH onto alumina column, discarding effluent.	This removes most of any residual Mo^{99} left on the alumina.
14.	Run 50 ml 0.01 <u>N</u> NH_4OH onto alumina column, discarding effluent.	
15.	Run 95 ml 3 <u>M</u> NaOH onto alumina column, discarding the first 15 ml of effluent and transferring the remainder to the Te^{132} product collection vessel.	
16.	Wash alumina with 20 ml H_2O.	Combine wash with Te^{132} product from preceding step.
17.	Transfer Te^{132} product from collection vessel to repurification vessel, maintaining air pressure under filter frit in latter vessel.	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.
18.	To product in repurification vessel add 60 ml conc. HCl containing 1 mg Te carrier.	
19.	Switch from compressed air to gaseous SO_2 under filter frit, allowing SO_2 to bubble through solution above frit until precipitation is complete.	SO_2 reduces Te to the element (black precipitates). Complete precipitation requires ~5 min.
20.	Switch from compressed SO_2 to partial vacuum under filter frit, filtering Te, and diverting filtrate to waste.	Use only as much vacuum as needed (250 - 400 torr).

21.	Readmit SO_2 gas under filter frit. Then add 100 ml H_2O, allowing SO_2 to bubble through it.	This washes the Te precipitate. Te is less soluble in water saturated with SO_2 than in pure H_2O.
22.	Repeat steps #20, #21, and #20.	
23.	Apply compressed air under filter frit and add 5 ml 10 N HNO_3.	This will dissolve Te.
24.	When no more Te is visible add 5 ml H_2O and wait 5 min.	This will dissolve any traces of Te not dissolved in step #23.
25.	Add 40 ml H_2O and store or load I^{132} generator (see Iodine-132). Alternatively, go to step #25a.	Solution is now 1 N HNO_3 and is ready for loading I^{132} 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.
25a.	Make 1 M in NaOH before shipping solution per se.	This prevents emanation of radio-iodine in transit. Any precipitated Te can be redissolved again in HNO_3 at destination.

Xenon-128

Isotope:	Xe^{128}
Shipping Form:	Gas sealed in glass ampoule
Half-Life:	∞ (stable)
Production Method:	$I^{127}(n,\gamma)I^{128} \xrightarrow[\tau\ =\ 25\ m]{\beta^-} Xe^{128}$
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:	>>95% 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 readsorption.

3. In steps #7, #9, #11, #14, and #16, substitute dry ice-solvent for liquid N_2, because Xe does not require as low a temperature as Ar for adsorption.

4. Some 8.9-day Xe^{129m} is also formed by $Xe^{128}(n,\gamma)Xe^{129m}$, 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:	$U^{235}(n,f)Sr^{90}$ + ...; (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:	$<10^{-8}$ mC Sr^{90}/ml; (note, therefore, that at radioactive equilibrium Sr^{90} is $<10^{-6}$% of Y^{90}).
Method of Analysis:	Chemical separation and β^- counting Sr^{90}; rapid assay possible prior to use of milked Y^{90} (described below).
Radiations (MeV):	Beta (Y^{90}): 2.26 Gamma (Y^{90}): None Beta (Sr^{90}): 0.54 Gamma (Sr^{90}): None
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.	Use 50 - 100 mesh resin.
2. Wash resin with 200 ml H_2O flowing upward at ~4 ml/min.	This removes excess H^+ from resin. Upward flow is more rapid than downward.
3. Pass 800 ml 2 N NH_4Cl over resin at ~2 ml/min.	This converts resin to NH_4^+ form.
4. Pass 1000 ml H_2O over resin.	This removes excess NH_4Cl.

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.	See Figures 12 and 13. Spaces are free of resin but filled with liquid and permit expansion of resin during chemical loading and washing.
2. Cap off both resin fill tubes and vent, leaving vessel entirely filled with liquid.	For fill tubes use small rubber "multi-injection" serum bottle stoppers. For vent use Tygon sleeve.
3. Connect product delivery tube to stopcock via Tygon connections.	Delivery tube should swivel freely and retract when sliding cover closes.
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.	Use a length of flexible plastic tubing compressed to fit.
7. Screw down top Lucite cover to seal against gasket.	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.	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.
2. Dissolve in required amount of distilled H_2O.	
3. Set aside ~5% of solution in a separate container.	Save for "back-titrating" in case of overshooting pH.
4. Using conc. NH_4OH, raise pH of main portion of citric acid solution to 5.4 - 5.5.	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 Sr^{90} to migrate, eventually ruining generator.
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.	See below for analysis to be performed prior to shipment.

D. Chemical Loading of Generator

1. Adjust flow rate through generator to ~3 ml/min.	Stopcock has screwdriver-operated Teflon needle valve for permanently setting desired flow rate when cock is full open.
2. Place generator in "clean" compartment of glove box, lining up eluent addition port with hole in ceiling of compartment.	Compartment isolates generator from Sr^{90} storage and dispensing equipment to prevent surface contamination of generator.
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.	

5. Add required amount of Sr^{90} stock solution slowly.	Use dispensing buret. Prevent solution from approaching gasket. Note: If ≤ 100 mC Sr^{90} are loaded, generator life will be $\sim$1 year (before Sr^{90} contamination in product begins to increase); if >100 mC are used, life will be shortened, presumably due to radiation damage of resin.
6. Wash funnel and generator with 3 30-ml portions of H_2O, allowing generator to drain after each washing.	Radiation level at one foot from generator will be $\sim$6 mR/hr for 100 mC loading.
7. Wash funnel and generator with 2 50-ml portions of citric acid preparation, allowing generator to drain after each washing.	
8. Remove funnel. Close hole in ceiling of "clean" compartment.	
9. Add enough powdered quartz (50 - 100 mesh) to fill re-entrant well over resin. Use citric acid preparation to wash quartz into place.	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.
10. Check flow rate of generator. If necessary readjust to $\sim$3 ml/min.	
11. Pass 2000 ml citric acid preparation through the generator.	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.

E. Milking Generator

1. Pour 100 ml citric acid preparation into top of generator through funnel.	This will milk off whatever Y^{90} has grown in since last milking. Knowing initial Sr^{90} activity, age of generator, and time since last milking, consult graph (Figure 14) to see how much Y^{90} is available.

2.	Place 125 ml flask (see note →) 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 β^- activity (Y^{90}).
2.	Add 10 ml saturated Na_2CO_3, 5 ml conc. NH_4OH, and 1 - 2 drops phenolphthalein.	
3.	Heat to near boiling for 30 min. with occasional stirring.	$SrCO_3$ should precipitate. If solution turns colorless add more NH_4OH.
4.	Cool in ice bath for 30 min.	
5.	Centrifuge, decant, wash precipitate with distilled H_2O, centrifuge, and decant.	Discard supernates.
6.	Dissolve precipitate in 5 ml 1 N HCl and heat for 15 min.	Use steam bath.
7.	Cool and add 1 - 2 drops phenolphthalein and 3 - 4 mg Y^{+++} carrier.	
8.	Titrate with 1 N NaOH to first sign of pink color.	
9.	Make up volume to ~20 ml.	Use distilled H_2O.
10.	Let stand 5 - 10 min. with occasional shaking. Centrifuge.	$Y^{89,90}(OH)_3$ precipitates.
11.	Decant supernate into 3 - 4 mg Y^{+++} carrier and 1 - 2 drops 1 N HCl. Mix well.	If solution is still pink, add HCl dropwise until colorless.
12.	Repeat steps #8 - #11 three times. Then repeat steps #8 - 10.	
13.	Decant supernate into 3 - 4 mg Fe^{+++} carrier and 1 - 2 drops 1 N HCl. Mix well.	

14. Titrate with 1 N NaOH and add 2 drops excess.	
15. Let stand 10 - 15 min. Centrifuge.	
16. To decanted supernate add 10 ml saturated oxalic acid solution and heat to near boiling.	
17. While hot, add conc. NH_4OH dropwise with stirring until a precipitate just forms. Digest hot for 15 min. with occasional stirring.	$SrC_2O_4 \cdot H_2O$ (strontium oxalate monohydrate) precipitates.
18. Cool in ice bath for 15 min. with occasional stirring.	
19. Filter onto pre-weighed filter paper.	Use ~15 mm diameter paper on a Hirsch funnel.
20. Wash precipitate with distilled H_2O, 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.	Use no desiccant.
22. Slowly admit air to desiccator.	
23. Repeat steps #21 - #22.	
24. Weigh and calculate percent recovery of Sr^{++}.	
25. Mount precipitate and count β^-'s, with and without 215 - 220 mg/cm^2 aluminum absorber.	This absorber removes all Sr^{90} β^-'s, leaving only the background from residual Y^{90}.
26. Correct count through the absorber for attenuation of Y^{90} β^-'s by the absorber.	Attenuation factor must be determined under identical conditions (backing, cover, thickness, area, position in counter) used for count in step #25.

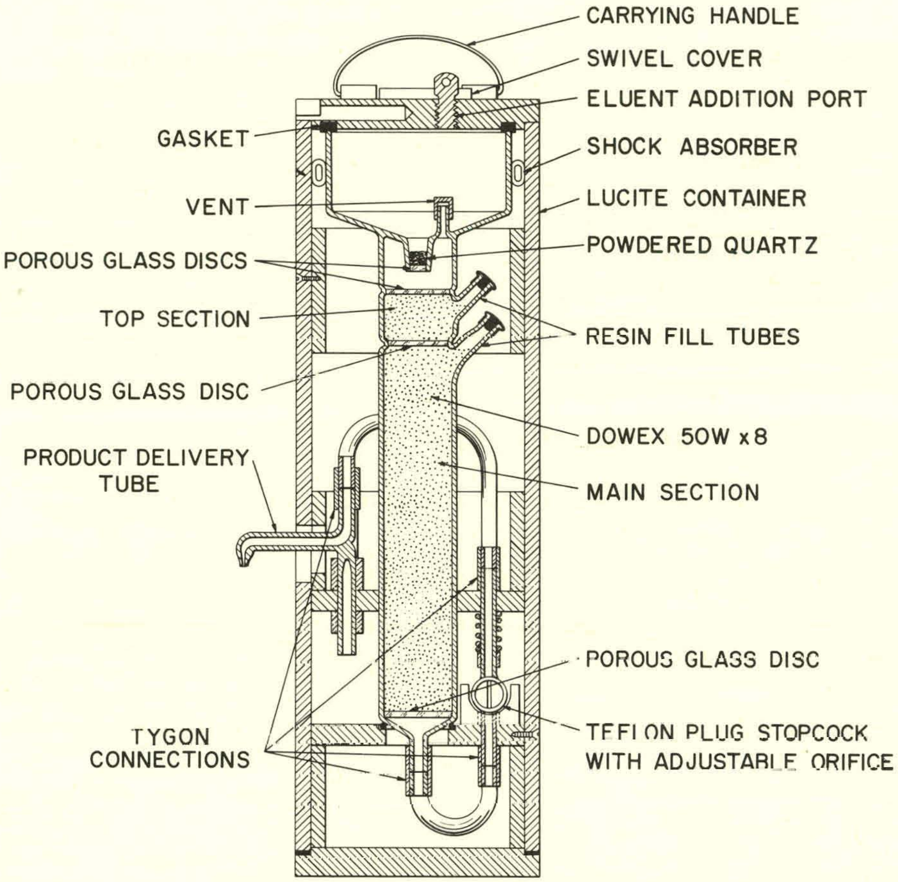

Figure 12. Yttrium-90 generator.

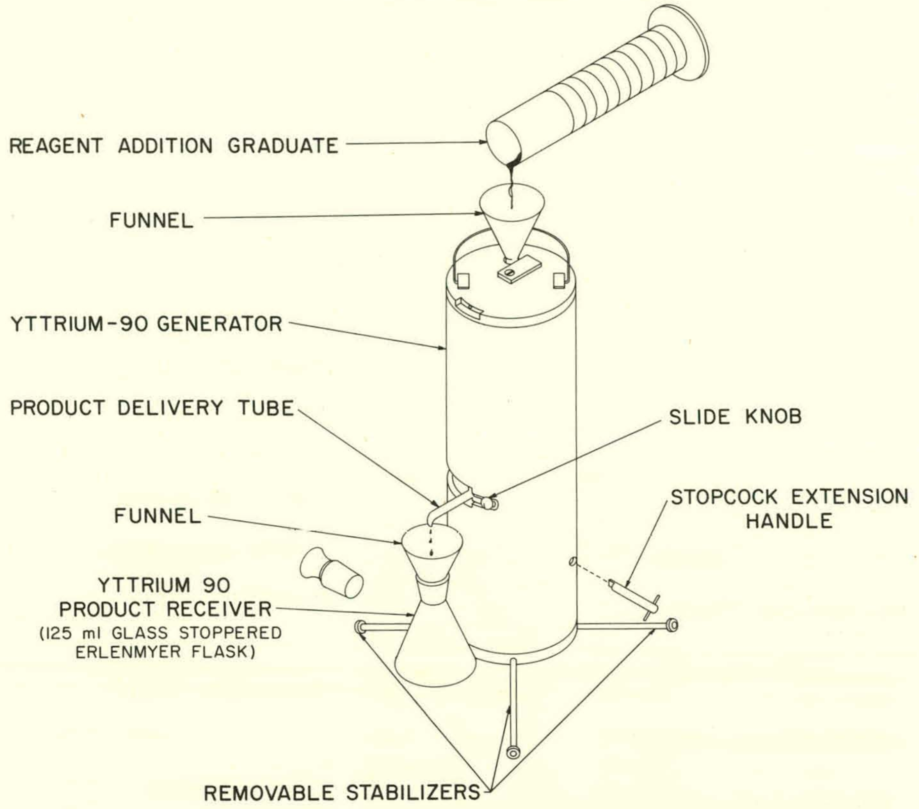

Figure 13. Operation of yttrium-90 generator.

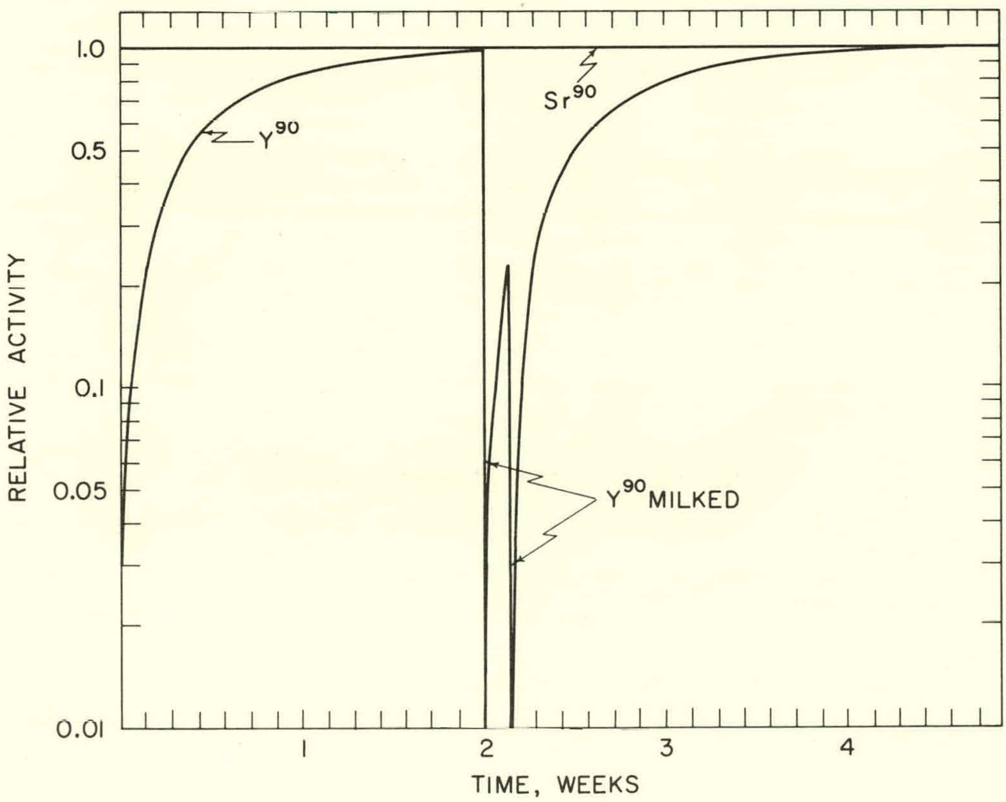

Figure 14. Curve showing growth of Y^{90} from its Sr^{90} parent and the effects of milking 2 weeks after and 1 day after previous milkings.

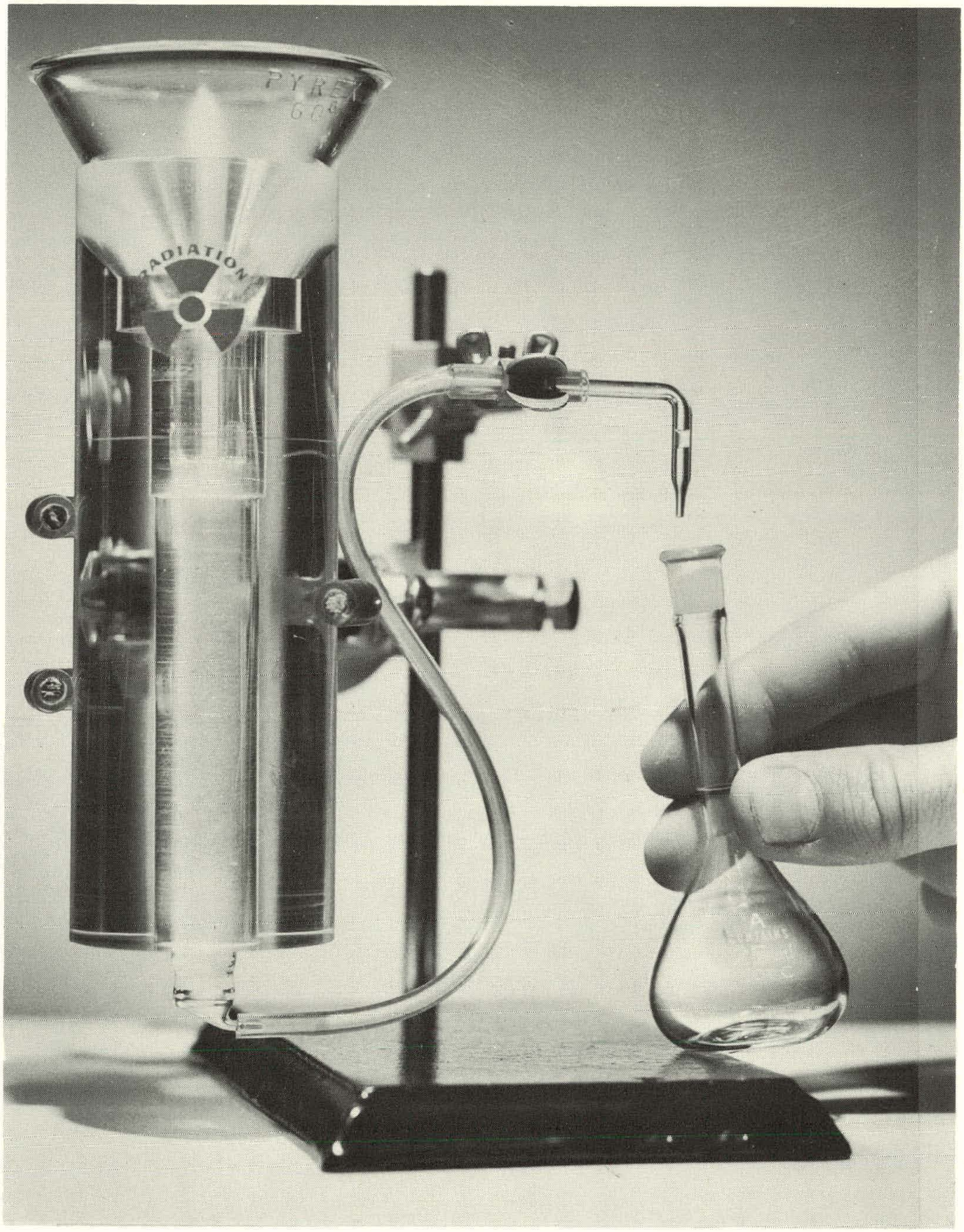

Figure 15. Strontium-90 assay column.

7. 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^{++} carrier [$\sim$0.1 M $Sr(NO_3)_2$], and Dowex 1 resin are furnished with each generator. See photograph (Figure 15).
2. Wash resin with 10 ml H_2O and adjust flow rate to $\sim$2 ml/min.	Raise or lower delivery tube as required.
3. Add 1 ml Sr^{++} 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} M NH_4OH until 25 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 μC 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.