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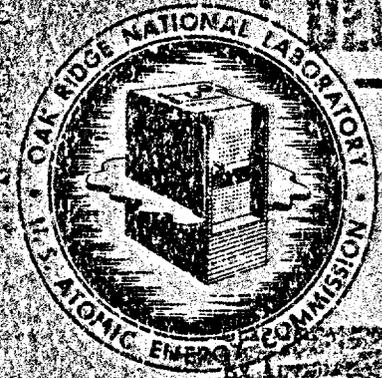
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ORNL
LABORATORY

ENRICHMENT OF URANIUM 236

By

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G. D. Murphy 43462
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Laboratory Research Dept.
1951

OAK RIDGE NATIONAL LABORATORY
OPERATED BY
CARBIDE AND CARBON CHEMICALS COMPANY
A DIVISION OF UNION CARBIDE AND CARBON CORPORATION
OAK RIDGE, TENNESSEE

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ENRICHMENT OF URANIUM 236

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ELECTROMAGNETIC RESEARCH DIVISION

Robert S. Livingston, Director

December 1951

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Oak Ridge, Tennessee

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Isotope Separation

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ABSTRACT

Neutron-irradiated uranium was electromagnetically separated to provide enrichment of the isotope of mass 236. The final isotopic abundance of U 236 was determined to be 95% in 1800 milligrams of uranium. Useful by-products of the program were 22 grams of 25% U 236 and 1100 grams of 99.7% U 235. These enriched materials have been made available for use on Atomic Energy Commission projects. Possible application of this separation process to recovery of the fuel from the MTR reactor is described.

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ENRICHMENT OF URANIUM 236

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One of the problems in the field of reactor development concerns the effect of the U 236 formed in the fuel assemblies as U 235 is consumed. In the thermal energy range, 15 percent of the neutron absorption in U 235 results in formation of long-lived U 236 (2.46×10^7 yrs). U 237 is formed by neutron capture in U 236 and is, therefore, proportional to the amount of U 236 present. U 237 decays to long-lived Np 237 (2.25×10^6 yrs) with a half-life of 6.7 days, emitting a 0.5 Mev beta and 0.27 Mev gamma. The neutron capture cross section for U 236 is of importance for reactors using enriched uranium requiring periodic reprocessing for chemical decontamination. The presence of U 237 activity, which is, of course, chemically inseparable from other uranium isotopes, determines the cooling time for partially expended fuel assemblies prior to the refabrication process. The nuclear characteristics of U 236 may also have a pronounced effect on the future management of uranium stocks depleted in the Hanford reactors. Diffusion plant processing of Hanford waste to provide material for atomic weapons would raise the U 236 concentration to about one-half percent. Even higher U 236 levels will result from prolonged irradiation with enriched fuel. The importance of carefully evaluating the nuclear properties of U 236, i.e., cross section, spontaneous fission rate, fission threshold, and related decay schemes has been clearly recognized. Consequently, it was decided to attempt separations to provide gram quantities of U 236 of as high purity as possible.

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The production and enrichment of U 236 was a cooperative effort involving the Hanford Plant, the Oak Ridge National Laboratory (X-10 and Y-12) and the Y-12 Plant. Approximately 2.5 kg of K-25 top product was irradiated for from 94 to 152 days in the Hanford reactors. The irradiated U-Al slugs were decontaminated at X-10 and shipped to Y-12 as nitrate solution. The uranium isotopes were separated in modified Beta-type calutrons operated by the Electromagnetic Research Division (ORNL) in Y-12. Product analyses and accountability control were provided by laboratories at Y-12.

The electromagnetic facilities used in the U 236 program included: two vacuum tanks in a magnetic field and related vacuum systems and electrical power supplies; five Beta-type calutrons with replacement parts for 45 runs; chemical wash area for removal of unresolved uranium deposits on equipment; chemical laboratory for extraction of wash solutions and preparation of charge material; and laboratory facilities for product recovery and purification.

SUMMARY OF U 236 SEPARATIONS

The enrichment of U 236 was undertaken in order to provide gram quantities of the highly purified isotope and to evaluate the potentialities of the electromagnetic process performance for this type of separation. To provide the desired product purity with 1% U 236 as source material, a two-stage separation process was required. The feed bank* contained 2200 grams of enhanced uranium, of which 23 grams was U 236. To obtain the desired quantity of feed for the second stage, a large fraction of the starting feed was separated during the initial stage, which incidentally isolated substantial amounts of highly enriched U 235. The disposition of feed and product

* Enriched Irradiated Uranium Feed Supply: Appendix B.

material through the two stages is as follows:

	Uranium gm	Isotopic Abundance		
		% U 235	% U 236	% U 238
Stage I: Feed Input	2200	92	1.0	6
U 235 Product	1125	99.7	0.1	0.1
U 236 Product	28	63	32	5
Unseparated	1085	76	1.2	21
Stage II: Feed Input	26	63	32	5
U 235 Product	5.2	98	2	0.2
U 236 Product	1.8	4	95.4	0.4
Unseparated	22	55	25	19

From intermediate level U 236 stocks, 26 grams of uranium were allocated to further enrichment. The remaining two grams were expended in mass analyses or were made available for distribution.

INVENTORY OF ENRICHED URANIUM 236*

Enriched Batch No.	Isotopic Composition** (weight percent)				Quantity on Hand (grams uranium)
	U 236	U 235	U 238	U 234	
2V-1	96.65	3.09	0.21	0.05	0.301
2V-5	95.51	4.00	0.43	0.06	0.210
***2V-2	95.31	4.18	0.36	0.15	0.230
2V-6	95.23	4.08	0.60	0.09	0.270
2V-3	94.90	4.63	0.40	0.07	0.119
2V-4	94.77	4.62	0.54	0.07	0.270
1V-0	42.38	53.80	3.16	0.66	0.294
1V-1	28.92	57.43	12.47	1.18	14.918
1V-2	18.49	58.06	22.36	1.09	1.217
1V-3	17.18	50.26	31.53	1.03	2.002
1V-4	14.16	44.52	40.44	0.88	1.963
1V-5	9.28	30.43	59.60	0.69	0.361

* Harmatz, B., and Livingston, R. S., Catalog of Highly Purified Uranium Isotopes, ORNL-1237, February 11, 1952.

** Limits of Error: Appendix H.

*** Spectrographic Analysis: Appendix I.

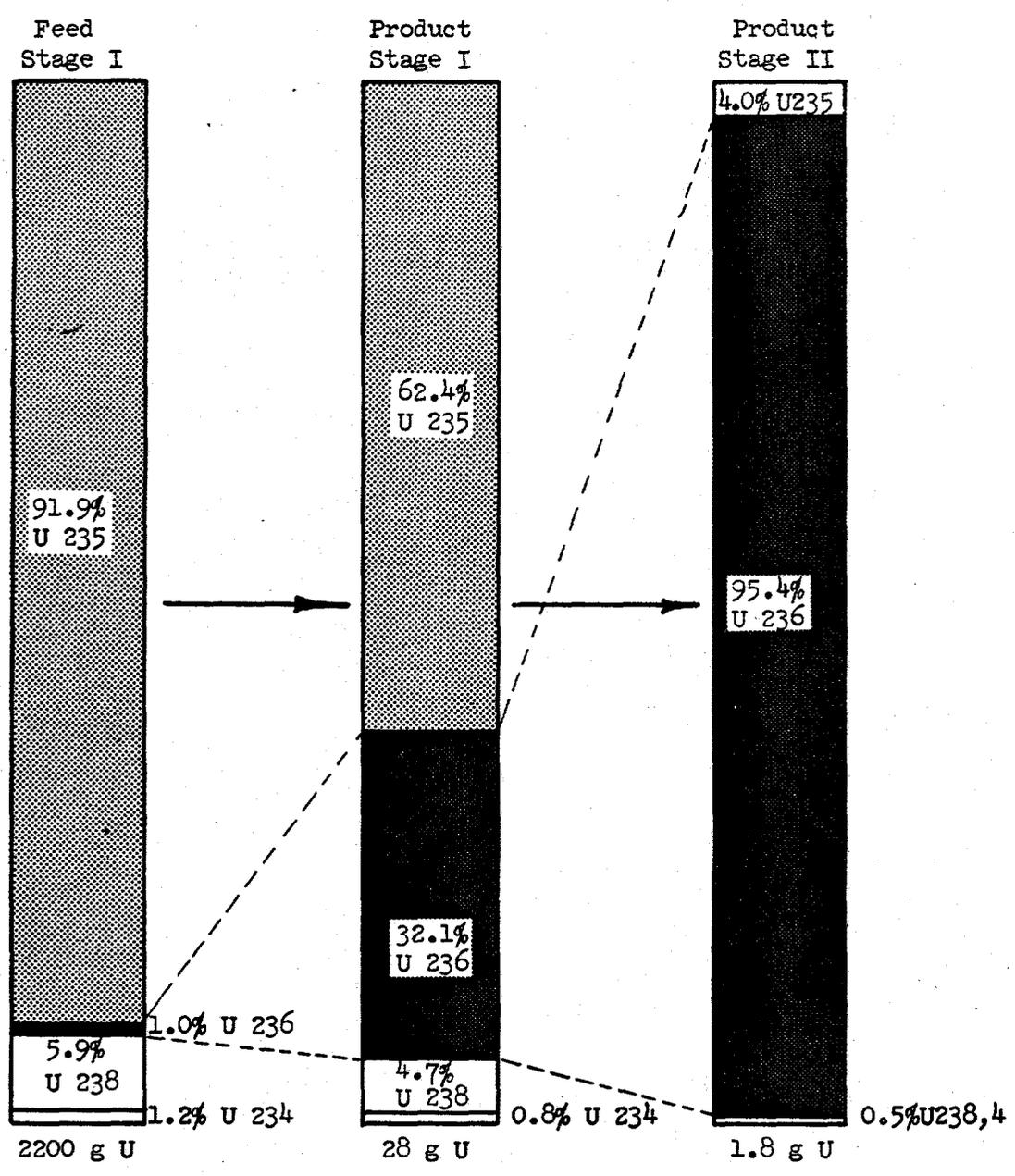


FIGURE 1. ISOTOPIC CONTENT OF STARTING FEED AND OF U 236 PRODUCTS

SHIPMENTS OF ENRICHED URANIUM 236

	Quantity (mg U)		Isotopic Content (%)		Proposed Determination or Use
	U 236	U 235	U 236	U 235	
Argonne National Laboratory	15.6	96.65	3.09		Fission experiments.
	10.2	95.31	4.18		U 237 beta decay
	7.5	42.38	53.80		Capture cross section for fast neutrons.
	5.0	42.38	53.80		U 236 and U 237 cross sections.
	3.0	37.60	58.74		Spontaneous fission rate, half life, and alpha energy.
K-25 Laboratory	30.0	37.60	58.74		Effect of U 236 presence on U 235 mass spectrometer assay.
Knolls Atomic Power Laboratory	20.0	42.38	53.80		Activation cross sections for thermal, intermediate neutrons.
Los Alamos Scientific Laboratory	150.1	95.31	4.18		Threshold detector of neutrons.
	10.0	37.60	58.74		Fission cross section at 2.5 and 14 Mev.
	10.0	37.60	58.74		Counting standards.
	153.1	94.90	4.63		Fission cross section versus neutron energy, inelastic scattering, fission fragment energy.
Oak Ridge National Laboratory	248.2	96.65	3.09		Absorption cross section, gamma-ray spectrum.
	203.0	35.28	61.26		Activation cross section.
	9.8	95.51	4.00		Fission cross sections.
University of California Radiation Laboratory	20.0	96.65	3.09		Specific activity.
	1.0	37.60	58.74		Systematics of alpha radioactivity.

DESCRIPTION OF THE ELECTROMAGNETIC PROCESS

THE U 236 MASS SPECTROGRAPH

The Beta calutron is in principle a Dempster-type mass spectrometer in which ions are accelerated by an electric field and are approximately focused after a deflection of 180° by the magnetic field, Figure 2. Isotope separation takes place in a vacuum tank evacuated to a pressure less than 0.03μ Hg. There is a 14-inch gap between the pole faces which form part of the vacuum chamber. Magnetic shims attached to the pole pieces reduce the focal spread resulting from angular width of the entering beam.

Solid uranium tetrachloride (UCl_4) is heated in the ion source to $\sim 500^\circ C$ to attain a suitable vapor pressure ($\sim 10 \mu$ Hg) for sustaining an arc. Uranium ions (principally U^+) are formed in the arc plasma which is generated by a beam of 150-volt electrons passing from a hot tantalum filament through a graphite chamber containing the UCl_4 vapor. The electron beam is shaped by a semi-elliptical defining slot $0.078''$ wide and $3/4''$ deep.

The positive ions formed in the arc are accelerated by a potential of 60,000 volts in the $1/2''$ gap between the arc chamber and the accelerating electrode. The apertures in both are approximately $3/8''$ by $7''$. In order to operate a convenient radius and magnetic field, ion energy is reduced to 36,000 volts by a decelerating electrode at ground potential; the arc chamber is at 36,000 volts positive.

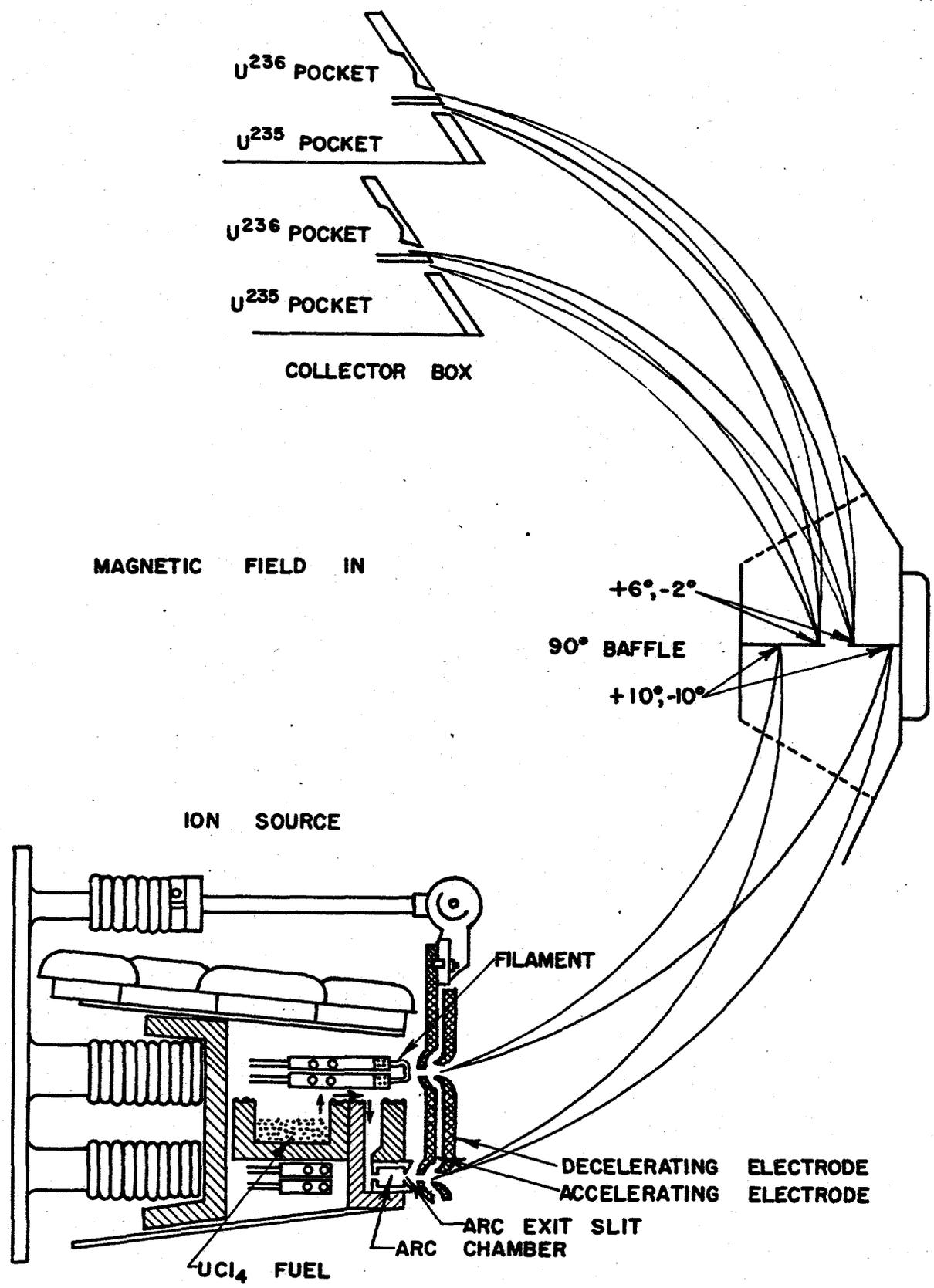


FIGURE 2. U^{236} MASS SPECTROGRAPH

The equation of motion of ions in the separation region is

$$V = 150 \left(\frac{e}{m}\right) \left(\frac{H}{c}\right)^2 r^2$$

where: $V = U^+$ acceleration = + 36 kv
 $e = U^+$ charge = 4.8×10^{-10} esu
 $m = U^+$ 235 mass = 3.9×10^{-22} gms
 $H =$ magnetic field = 6700 gauss
 $c =$ velocity of light = 3×10^{10} cm/sec
 $r =$ radius of U^+ particle = 61 cm

From the above constants and parameters, it can be shown that U 235-U 236 peak separation is 0.1 inch at 180° .

In the main separation region of the tank, the intensity of the U^+ beam is uniform about the normal trajectory up to $\pm 10^\circ$ angular variation.* To improve beam focus and collector life, angles of emergence from the source which are admitted to the collector are limited to a band of $+6^\circ$ to -2° by suitable baffling at the 90° point in the normal orbit.

Collector pockets for U 235 and U 236 ion beams are designed to permit U^+ 235 monitoring and a greater retention of ions arriving at the U 235 pocket. The U 234 and U 238 ion beams impinge on graphite defining plates, except in the case of certain four-pocket collector boxes. The standard asymmetrically-curved slot boundaries are reworked to a symmetrical shape to improve the fit of the ion focal pattern. Collector slots are of uniform 0.050" width, separated by a 0.060" defining edge. Ion beam erosion of the defining edge limits collector life to 30 separation hours. This short life problem was solved by using two collector units for each first-stage run.

The disposition of vaporized source material and of the different isotopes is illustrated in the flow diagram, Figure 3.

* In traversing radial paths from the ion source to the collector slot, positive angle beams travel less than 180° arcs, while negative angles take paths longer than 180° .

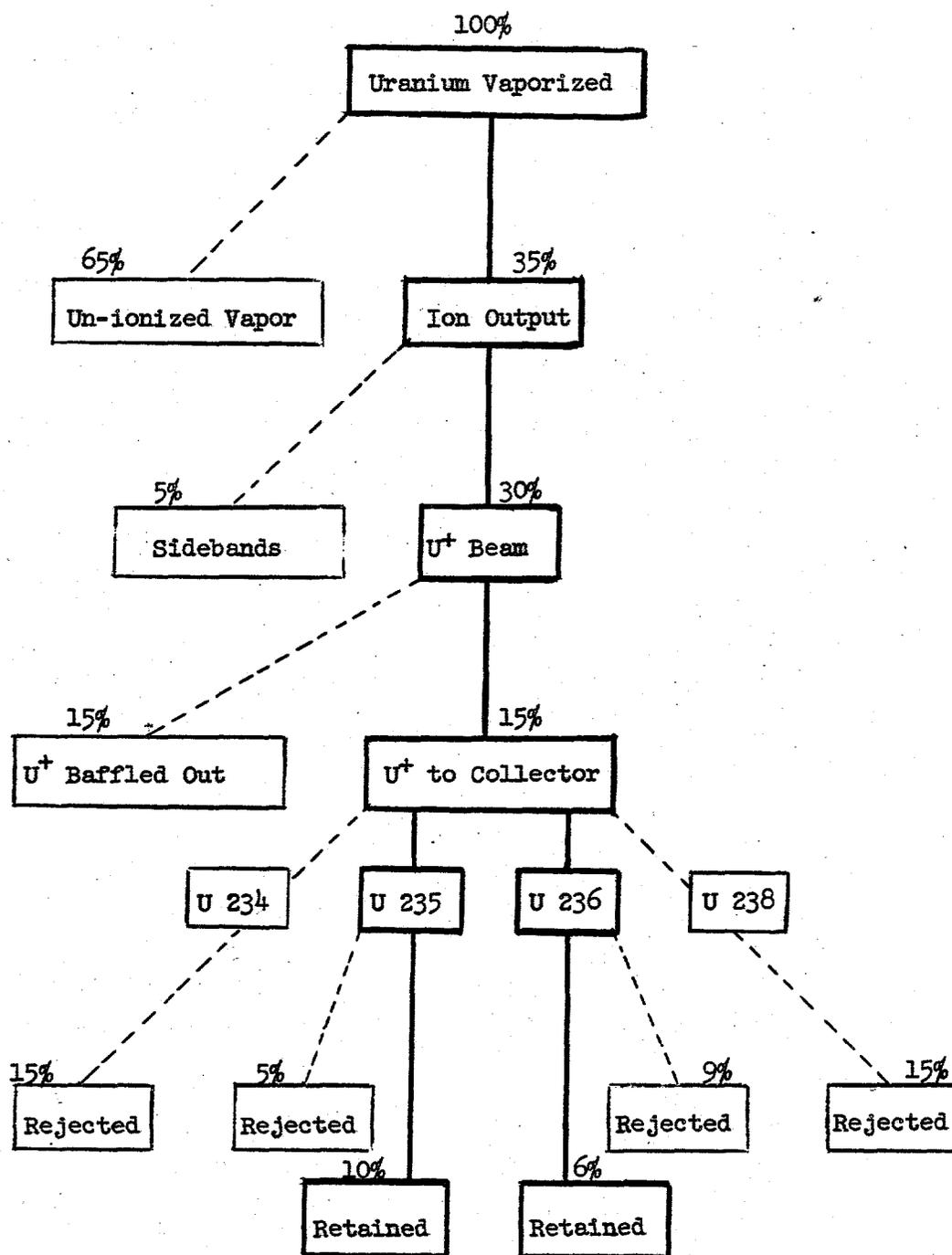


FIGURE 3. APPROXIMATE URANIUM DISTRIBUTION IN EQUIPMENT, FIRST STAGE

THE CHEMICAL PROCESS

The following four basic phases of operation of the Beta chemical process* also apply to the special separation described in this report:

1. Selective recovery of uranium deposited in graphite collector pockets.
2. Non-selective recovery of uranium deposits on metal, ceramic, and graphite surfaces of the calutron.
3. Extraction, purification, and conversion of enriched isotopes to U_3O_8 .
4. Extraction, purification, and conversion of unseparated material to UCl_4 .

Chemical processing facilities designed for enrichment of U 234** were applicable to the U 236 separation program. Quantitative recovery methods for recycling limited amounts of uranium were extended to batches of U 236 only one-half the size of U 234 batches.

On termination of a separation run, the calutron is removed from the magnet and the collector is withdrawn for removal of product materials. The U 236 collector pockets are vacuum-scraped and the scrapings are treated in a cycle involving muffling to an ash, leaching with nitric acid, filtering, and the standard penta-ether extraction to produce pure oxide. Care in disassembly and scraping of the U 236 pocket was found to make a significant improvement in the recovery efficiency and enrichment. The U 235 deposits are extracted in a parallel process.

* Chemical Processing Equipment: Electromagnetic Separation Process, National Nuclear Energy Series, Div. I, Vol. 12, 1951.

**Case, F. N., Facilities for Processing Alpha-Active Isotopes, Y-659, September 1, 1950.

Contaminated surfaces of the equipment are spray-washed in an enclosed stainless steel tank. A recirculating spray of nitric acid solution and water dissolves the bulk of the adherent uranium prior to disassembly and a final scrubbing. The uranium-bearing wash solution, about fifty gallons, is filtered and evaporated, acidity and specific gravity adjusted, and the uranium extracted with diethyl ether. A hydrogen peroxide precipitation effects a final purification and reduces the volume for chlorination. The uranium peroxide cake is dissolved in hydrochloric acid, re-evaporated to a chloride cake and converted to the tetrachloride by refluxing with liquid hexachloropropene as a chlorinating reagent. Contents of the chloride reactor are filtered into a stainless steel container, washed with CCl_4 and baked for several hours in a vacuum furnace. In preparing a small feed batch, the chief task is minimizing the reaction of hygroscopic UCl_4 with water vapor, forming UOCl_2 . Operation with the oxychloride is characterized by excessive temperatures, low output, and poor resolution.

Various salvage operations are carried out on graphite parts of the calutron and on insolubles from wash operations. These salvage items are muffled to an ash and combined with the bulk of the material before the ether extraction step. Condensates and effluents containing less than 0.05 ppm uranium are re-used as machine wash. The distribution of uranium throughput in the electromagnetic cycle is illustrated in the flow diagram, Figure 4.

It is established that there is uranium holdup in the vacuum systems, furnaces, evaporators, hoods, etc., which tends to increase with continuous recycling. The first stage of isotopic enrichment is closed with an intensive effort to recover holdup material, mainly to minimize the prospect of contaminating second-stage feed. Effective recovery of unresolved deposits on termination of the second stage would supply feed for subsequent U 236 enrichments.

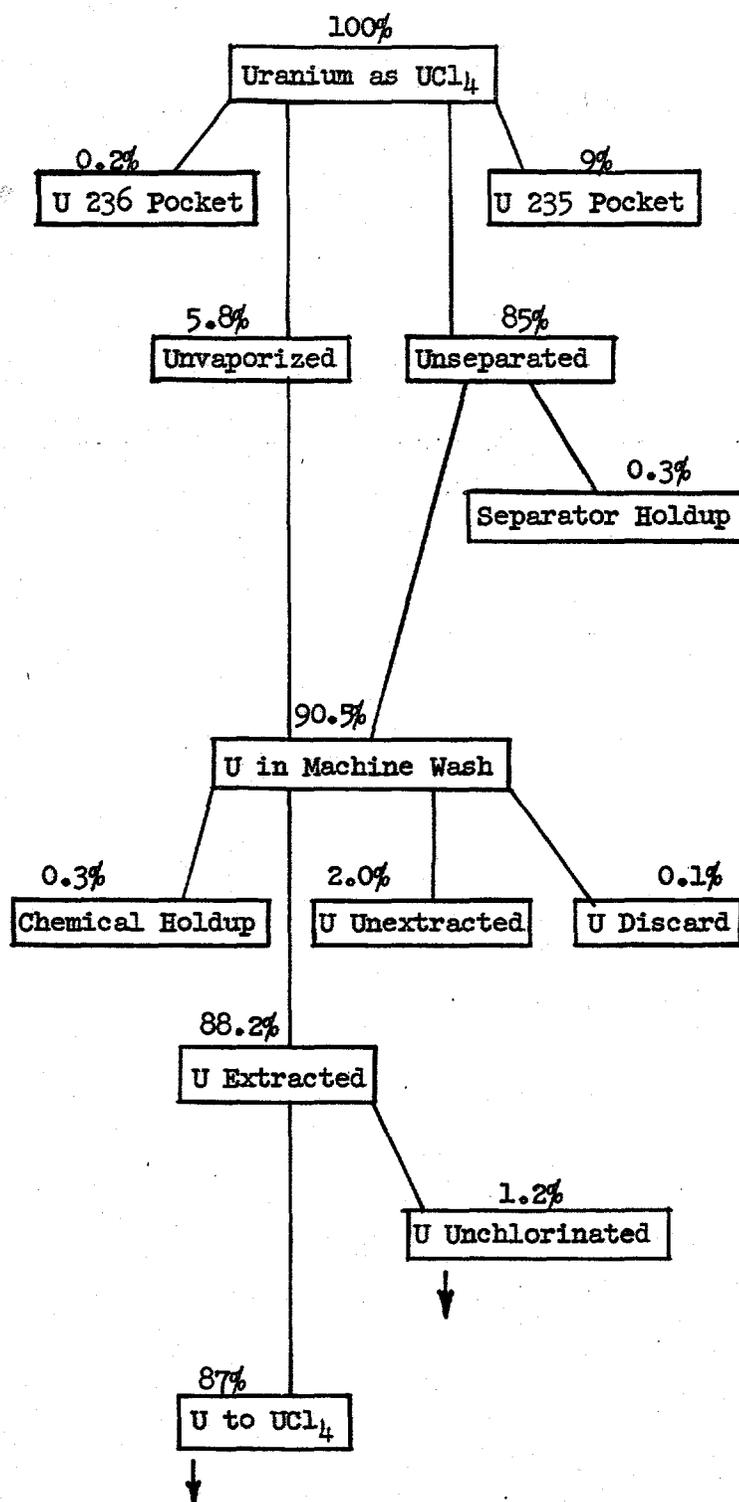


FIGURE 4. APPROXIMATE URANIUM DISTRIBUTION IN ELECTROMAGNETIC CYCLE, FIRST STAGE (~ 350 g U Feed per Run)

Operating specifications in the first stage provide for blending unseparated feed with sufficient make-up to maintain a uniform weight of source material. The feed-limited condition in the second stage precludes make-up, consequently decreasing yield per run is expected with elapsed cycling.

ELECTROMAGNETIC PROCESS PERFORMANCE STANDARDS

A second objective of the U 236 separation program was to evaluate the capabilities and limitations of electromagnetic equipment and methods. Process performance values are obtained through assay and analysis of feed and product batches. Derived performance standards are generally applicable to similar projects, e.g., proposed plutonium isotope enrichment.

Enhancement* factors are used to determine the isotopic composition of the product for various isotopic constituents of feed. Since the first stage U 236 product is enhanced 40-fold relative to U 235, the 236:235 product ratio is 40 times greater than the corresponding feed ratio.

<u>Product</u>	<u>Enhancement Relative to</u>			
	<u>U 236</u>	<u>U 235</u>	<u>U 238</u>	<u>U 234</u>
First Stage U 235	16	--	82	35
First Stage U 236	--	40	54	52
Second Stage U 235	26	--	71	26
Second Stage U 236	--	47	61	36
Overall U 236	--	1880	3300	1870

The overall enhancement in a multiple-stage separation process is the product of the single-stage enhancement factors.

* The general definition of enhancement is given in Appendix G.

Process efficiency is defined as the ratio of the collected isotopes to the vaporized isotopes and is a function of source and collector efficiencies. With the resolution listed in the table above, first stage gave 9.8% process efficiency for U 235; that is, 10% of vaporized U 235 atoms were transmitted to the collector pocket at which point 98% of the atoms arriving were retained.

<u>Product</u>	<u>Efficiency (%)</u>		
	<u>Source</u>	<u>Collector</u>	<u>Process</u>
First Stage U 235	10	98	9.8
First Stage U 236	10	62	6.2
Second Stage U 235	8	96	7.6
Second Stage U 236	8	63	5.0

Reduction of source performance for the second stage is attributed to the charge-limited condition.

Isotope production rate depends on separator process efficiency and on the rate of fuel vaporization. Productivity per run is governed largely by the limitation on weight of source material. For the first stage the separation period averaged 55 hours per run for a twin-arc ion source. The separation period of the single-arc source used in the second stage averaged 6.5 hours per run.

<u>Performance Parameters</u>	<u>Stage 1</u>	<u>Stage 2</u>
Fuel vaporization rate (gm U/hr-arc)	3.2	2.8
Fuel vaporized per run (gm U 235)	307	11.1
(gm U 236)	3.9	5.6
Collector current per arc (ma U ⁺)	35	25
Production rate per arc (mg U 235/hr)	273	128
(mg U 236/hr)	2.2	42
Production per run (gm U 235)	30	0.85
(gm U 236)	0.24	0.28

Chemical recovery efficiency pertains to the processing of the product deposits in the collector pockets and to the large unresolved fraction to be re-used as source material. The efficiency of isotope recovery is evaluated at the end of each stage as a percentage of the throughput and as a percentage of the starting inventory. Throughput recovery efficiency is independent of the number of recycles, whereas the unaccounted fraction of initial feed is expected to increase with elapsed cycling.

	<u>Isotope</u>	<u>Recovery Efficiency (%)</u>	
		<u>Throughput</u>	<u>Feed</u>
First Stage (9 cycles)	U 235	99.5	97.0
	U 236	99.6	97.5
Second Stage (6 cycles)	U 235	--	--
	U 236	97.0	87.5

The second stage recovery efficiency for U 235 is not valid because of U 235 pickup from process equipment.

Utilization efficiency is the index of feed conservation, particularly the U 235 and U 236 components. U 235 utilization efficiency increases with production efficiency and plant recovery and decreases with U 235 contamination in U 236 product. Thus, in the first stage, 93% of the U 235 fraction is converted to product, 1.6% is diverted to U 236 concentrate and 5.2% is dispersed.

	<u>Isotope</u>	<u>Utilization Efficiency (%)</u>		
		<u>Chemical Recovery</u>	<u>Collector</u>	<u>Overall</u>
First Stage	U 235	94.8	98.4	93
	U 236	94.0	90.7	86
Second Stage	U 236	61.3	94.5	59

About 10 percent of U 236 processed in the first stage is diverted to the U 235 collector pocket. Reseparation of this material would raise the U 236 utilization efficiency to the U 235 value.

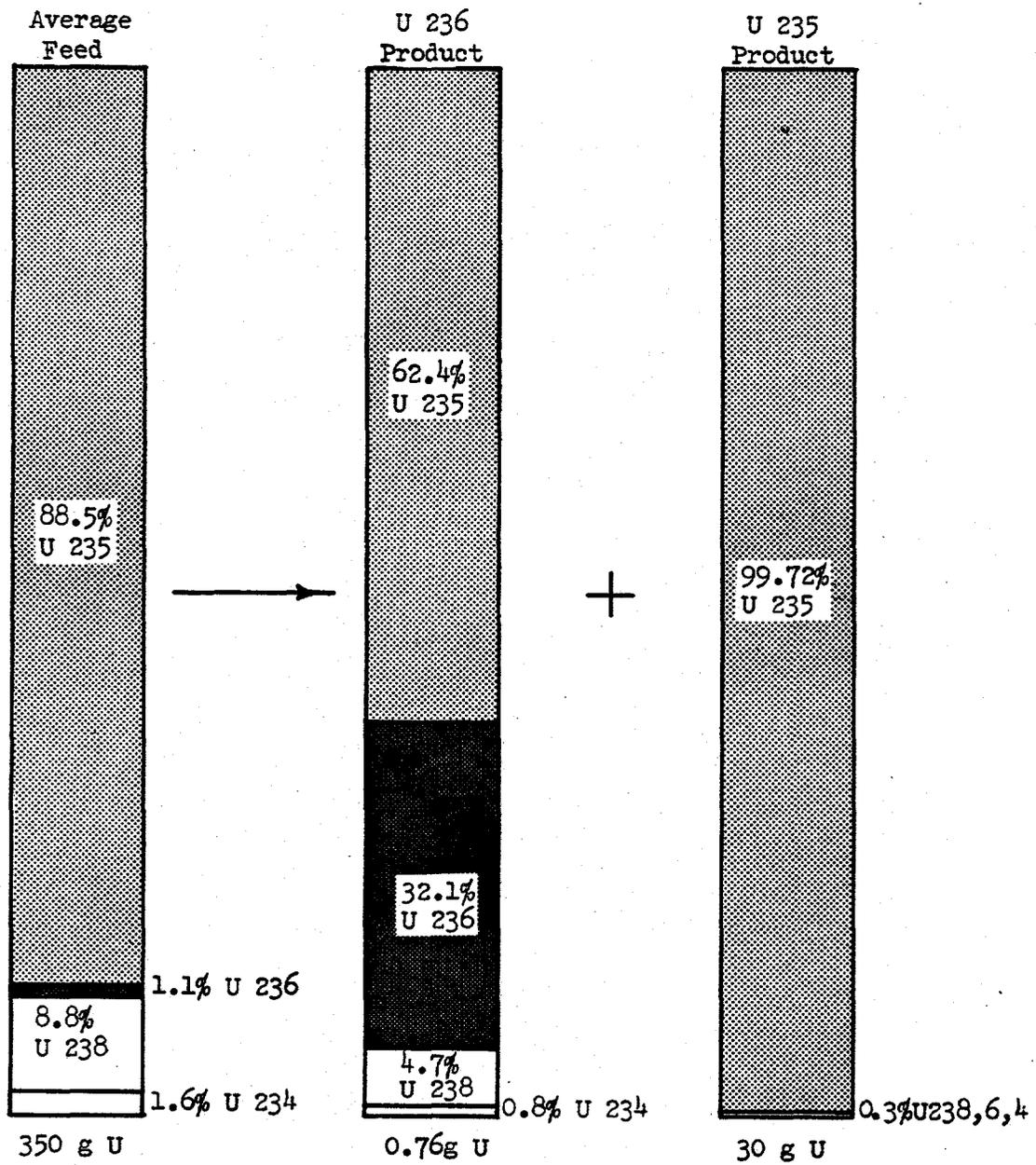


FIGURE 5. ISOTOPIC CONTENT OF FEED AND PRODUCT PER RUN - STAGE 1 (TWO-POCKET COLLECTOR)

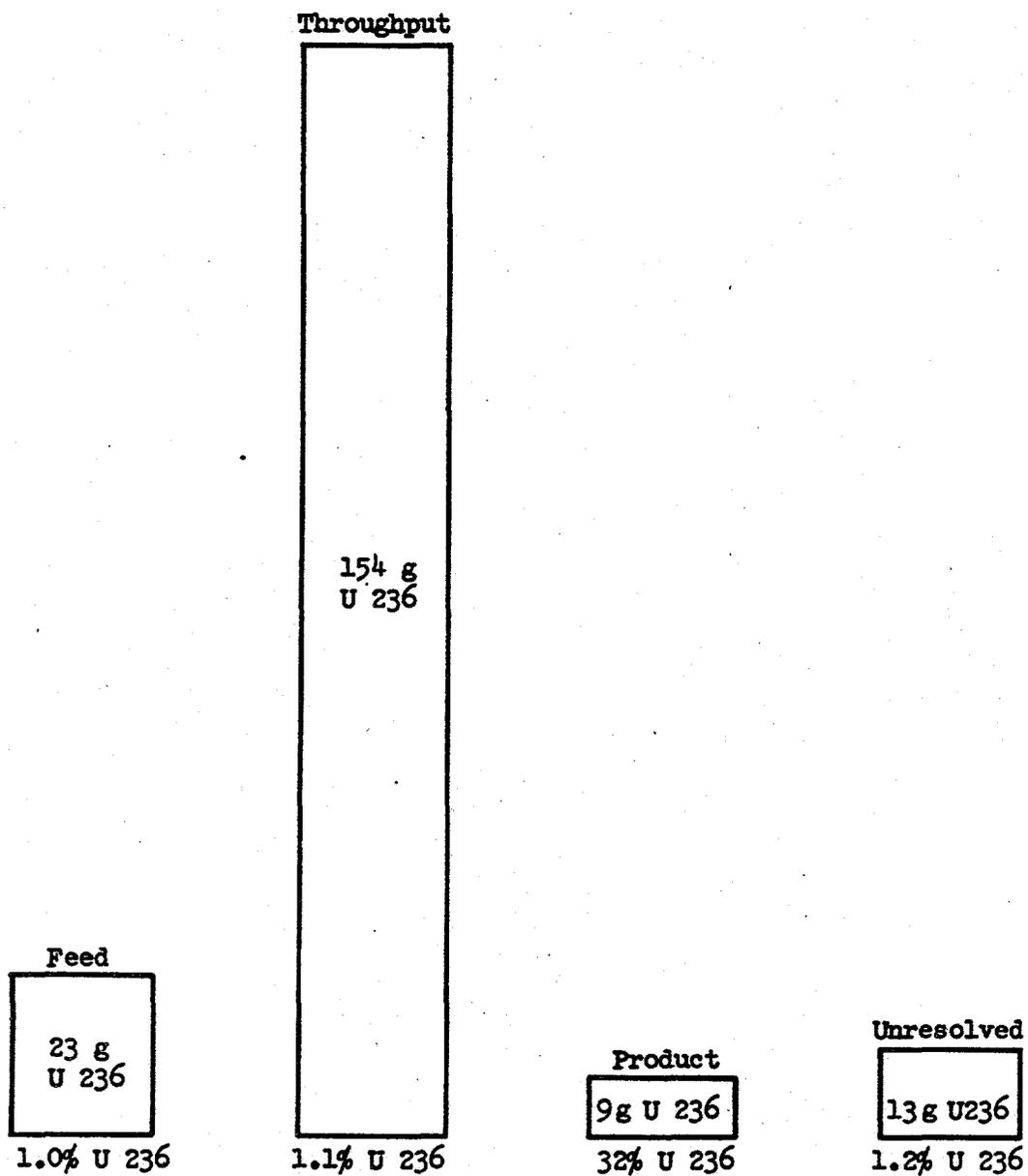


FIGURE 6. URANIUM 236 CONTENT IN FEED, THROUGHPUT, PRODUCT AND UNRESOLVED - STAGE 1

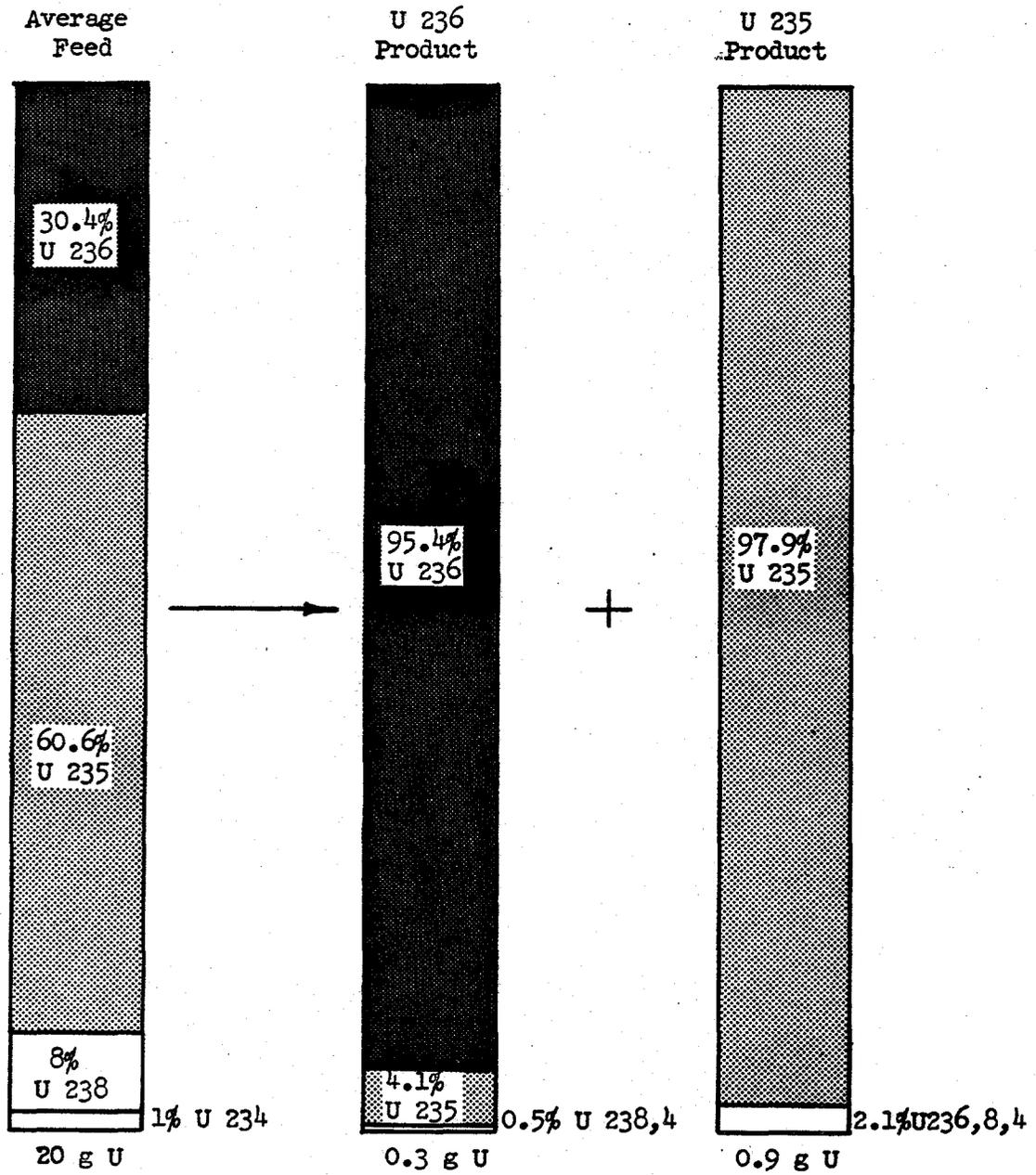


FIGURE 7. ISOTOPIC CONTENT OF FEED AND PRODUCT PER RUN STAGE 2

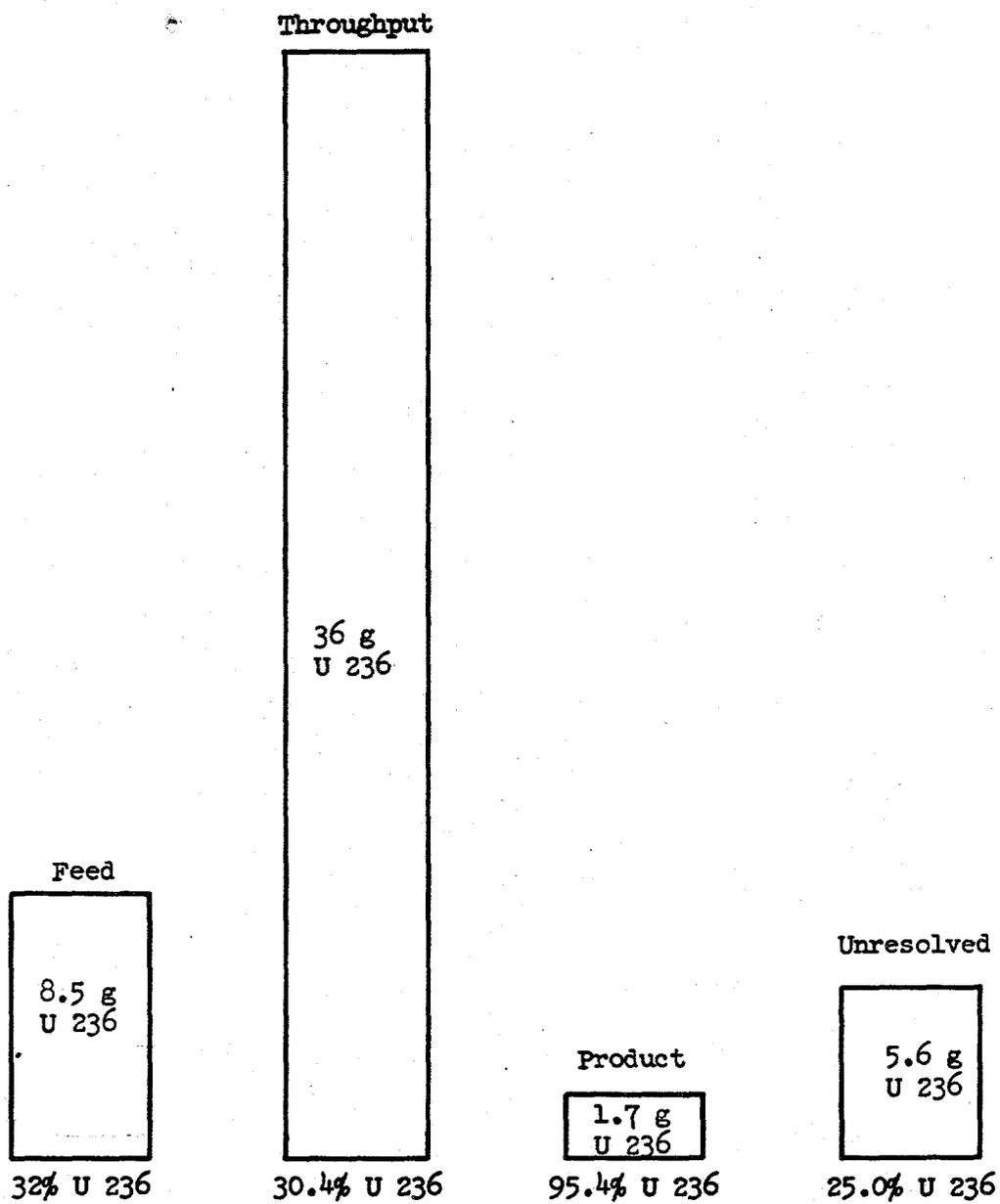


FIGURE 8. URANIUM 236 CONTENT IN FEED, THROUGHPUT, PRODUCT AND UNRESOLVED - STAGE 2

ANALYSIS OF PROCESS DESIGN AND BEHAVIOR

Feed Versus Product Purity

The isotopic composition of available source material determined the selected approach to the separation program. Recovery of uranium from irradiated fuel rods totaled 2 kg of U 235 which was divided batch-wise into seven parts in accordance with the criticality safe limit factor. It appeared feasible to segregate the rods according to time of irradiation in order to facilitate the enrichment of U 236 initially. Therefore, feed U 236 concentration at the start varied in a regular manner, being either 0.76% or 1.3% U 236. Inhomogeneity of fuel material resulted in discrete products of either 25% or 35% U 236.

Enrichment of U 236 was principally a separation from U 235 for two reasons: U 235 abundance in the starting material was quite large, about 92%, and resolution between U 235 and U 236 focal patterns was minimized by the single unit mass difference. Samples of U 236 highly depleted in U 235 were particularly desired for thermal capture cross section determinations. As a result of first stage processing, the ratio of U 236 to U 235 was lifted from 1/92 to 1/2. From the second stage separation, the corresponding ratio was raised from 1/2 to 25/1. The abundance of U 236 relative to both U 238 and U 234 was finally 200/1.

Variation in Isotopic Content of Recycled Uranium

On prolonged re-use of starting feed, the source material is changed isotopically, with subsequent effects on product concentration. Changes in the isotopic composition of reprocessed uranium depend on collector retention and enhancement factors and on the introduction of foreign uranium to the cycle.

The combined effect of non-uniform removal of isotopes in product and the pickup of uranium-bearing fractions is summarized in Figures 9 and 10.

The pick-up of foreign uranium during recycle recovery operations averaged 2.5 grams of U 238 per cycle during the first stage. Uranium diluent to the second stage is calculated to be 0.2 grams of U 235 and 0.18 grams of U 238 per cycle. The comparative collection of uranium isotopes is evaluated as follows:

<u>Isotope</u>	<u>First Stage</u>	<u>Second Stage</u>
U 235	9.9 %	7.7 %
U 236	6.7	5.3
U 238	0.25	0.19
U 234	0.45	0.43

Based on collector utilization of uranium ions, a depletion of U 235 in source material and an increase of other isotopic components is expected with elapsed cycling.

On termination of each of the two stages, an effort was made to recover all uranium contained on electromagnetic equipment and in salvageable residues. Cleanup and salvage processing yielded considerable uranium somewhat diluted by U 238 as indicated in the following table:

	<u>Terminal Recovery</u>	<u>Isotopic Composition (%)</u>				<u>Uranium (gm)</u>
		<u>U 235</u>	<u>U 236</u>	<u>U 238</u>	<u>U 234</u>	
First Stage:	Source Feed	81	1.3	15	2.5	686
	Holdup and Salvage	66	1.0	31	1.7	400
Second Stage:	Source Feed	57	29	13	1.2	15.0
	Holdup and Salvage	50	18	32	0.9	7.5

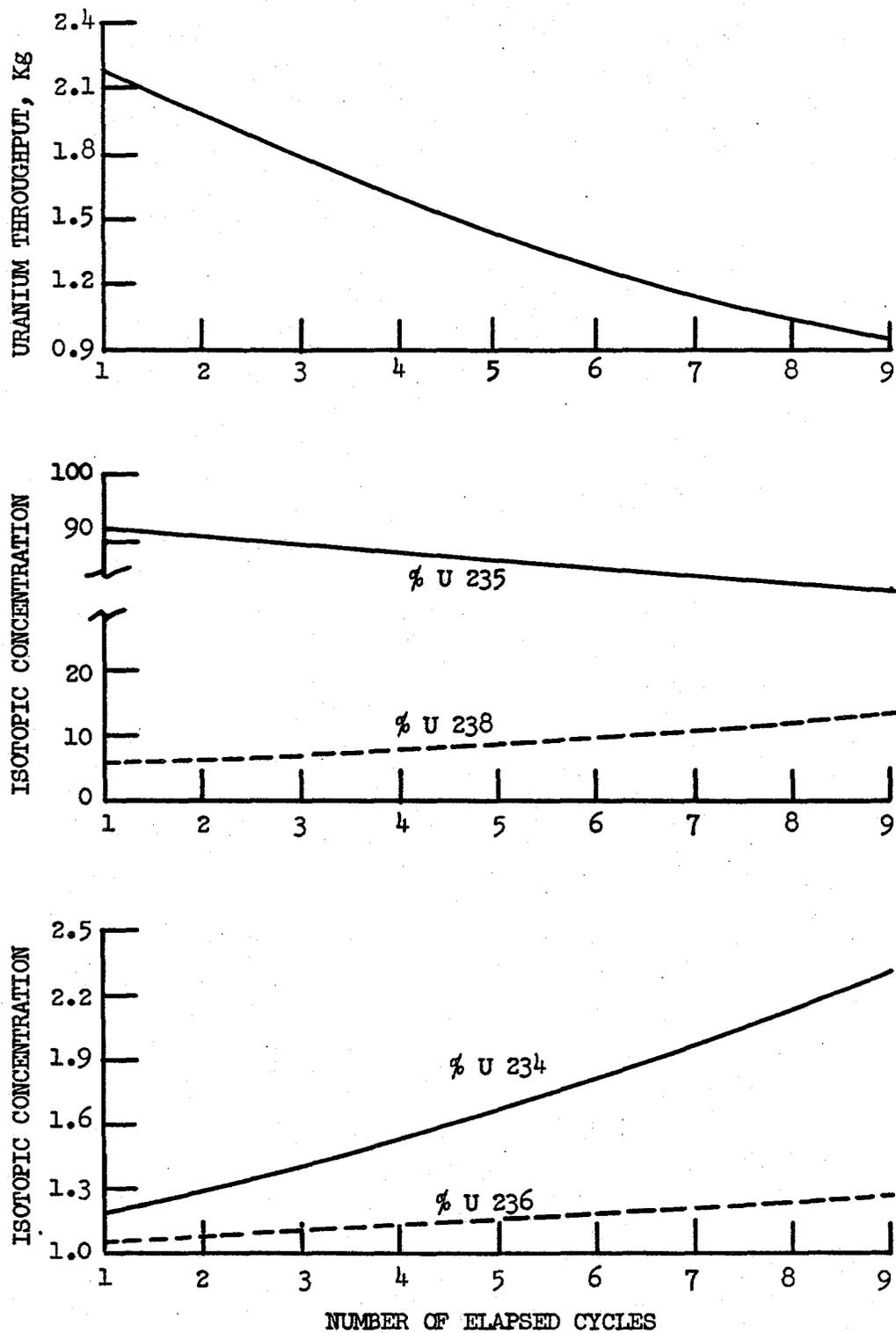


FIGURE 9. QUALITY AND QUANTITY OF URANIUM THROUGHPUT PER CYCLE - STAGE 1

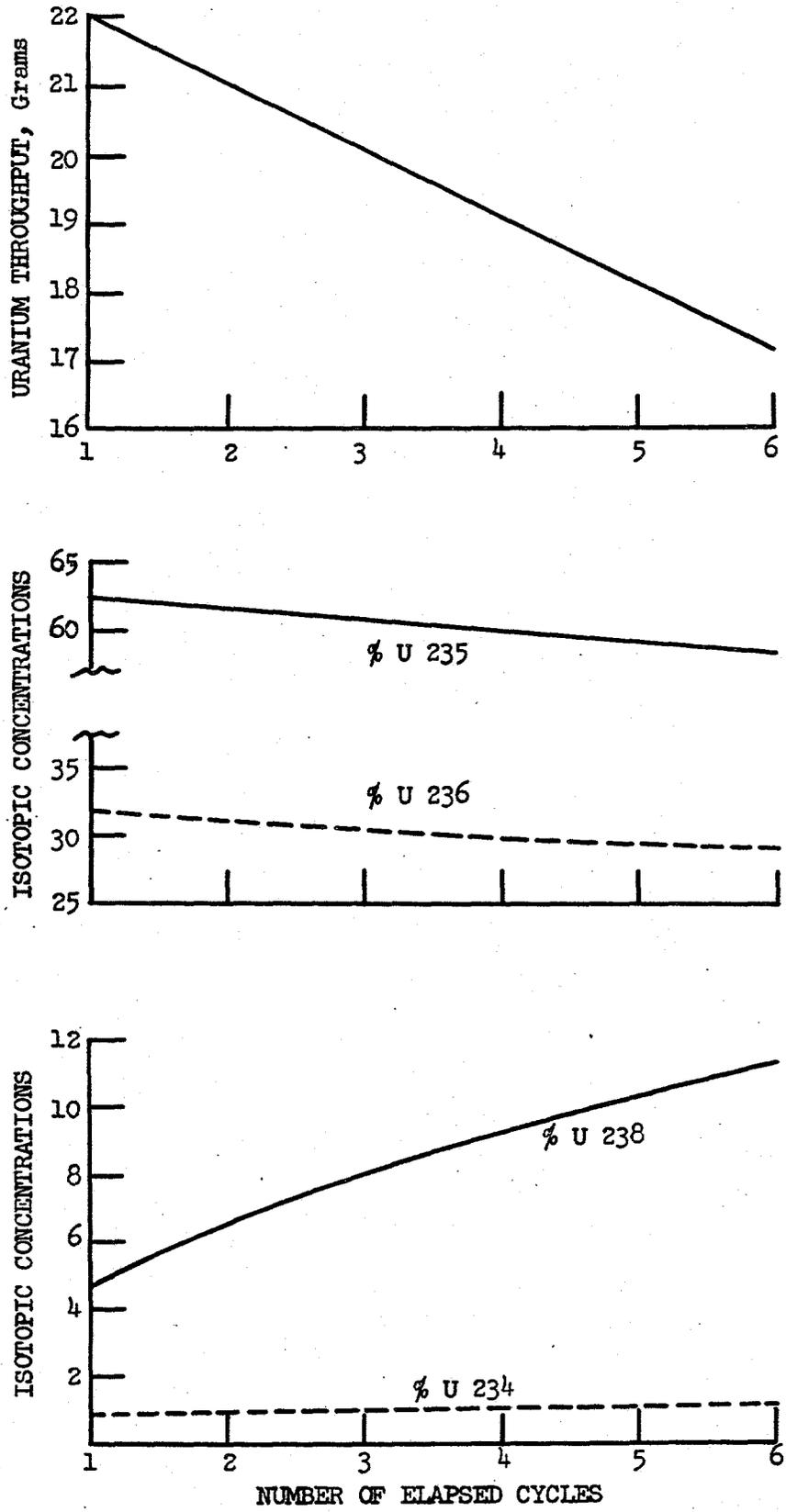


FIGURE 10. QUALITY AND QUANTITY OF URANIUM THROUGHPUT PER CYCLE - STAGE 2

Properties of Isotopic Foci

At the collector slots ion current density distributions due to the four isotopes are the same except for an intensity factor and relative displacement. The ion current distribution due to a single isotope exhibits a skewness towards the next lower mass. Mainly because of the asymmetrical shape of the focal spread, upper pocket product is enhanced nearly two-fold relative to lower pocket material for a relative displacement of one mass-unit. Since the entrance slit width of 0.05" approximates the focal sharpness, utilization of the ion beam by the collector is considered adequate. Thus, 0.06" of space of the 0.11" dispersion between U 235 and U 236 peaks remains for a defining edge. The defining edge is a target for unresolved "valley" current and will withstand ion impact for as long as 30 hours.

Focal widths respond especially to fine adjustment of accelerating voltage, arc voltage and current, and vapor pressure. A vertical adjustment of the accelerating slit is indispensable for improving intensity of the most favorable angular distribution. The exact fit of isotopic foci to movable entrance slits requires continuous monitoring of the resonance voltage and periodic visual observation of the focal pattern. Effective completion of the separation process requires extracting the divided isotopes from the graphite collector pockets with great care.

Separator Development

Within the limits imposed by uncertainty of performance, U 236 separator geometry was based on contemplated production capacity and enhancement requirements. In the first place the aspect of the beam emerging from the source at all angles between $\pm 10^\circ$ with uniform density per degree must be considered. The choice of utilized beam is required to meet both resolution and collector

life objectives. Fortunately, the two factors are to some extent compatible and an 8° angular band was adopted after preliminary tests. The boundary for the prescribed range is a compromise between U 236 pocket retentivity and U 235 pocket monitoring, which are somewhat incompatible factors. Baffles at the 90° point of the normal orbit were installed to transmit only angles of emergence from the source in the range of -2° to $+6^\circ$. Width and shape of U 235 and U 236 entrance slits remained to be determined. The entrance was narrowed to 0.05" and the conventional Beta slot boundary was modified to fit the given isotopic foci, Figure 11a.

An experimental four-pocket collector, Figure 11b, was tested during the first stage for the collection of mass fractions 234, 235, 236 and 238. Each of the curved collector slots is of uniform 0.05" width and is displaced from adjacent slot openings by graphite defining edges. The results obtained from this collector are summarized in Figure 12. Enhancement and retention parameters for each of the four pockets are as follows:

Principal Isotope	Enhancement Relative to				Retention Efficiency (%)
	U 238	U 236	U 235	U 234	
U 236	56	--	40	72	62*
U 235	90	16	--	29	98
U 234	56	44	30	--	61
U 238	--	68	210	250	72

Insertion of U 234 and U 238 collectors lowered the intensity of scattered components in the direction of U 236 and U 235 slots as indicated by improved U 236:U 234 and U 235:U 238 enhancements.

* U 236 pocket retention efficiency was not cleanly evaluated in the presence of a U 238 pocket.

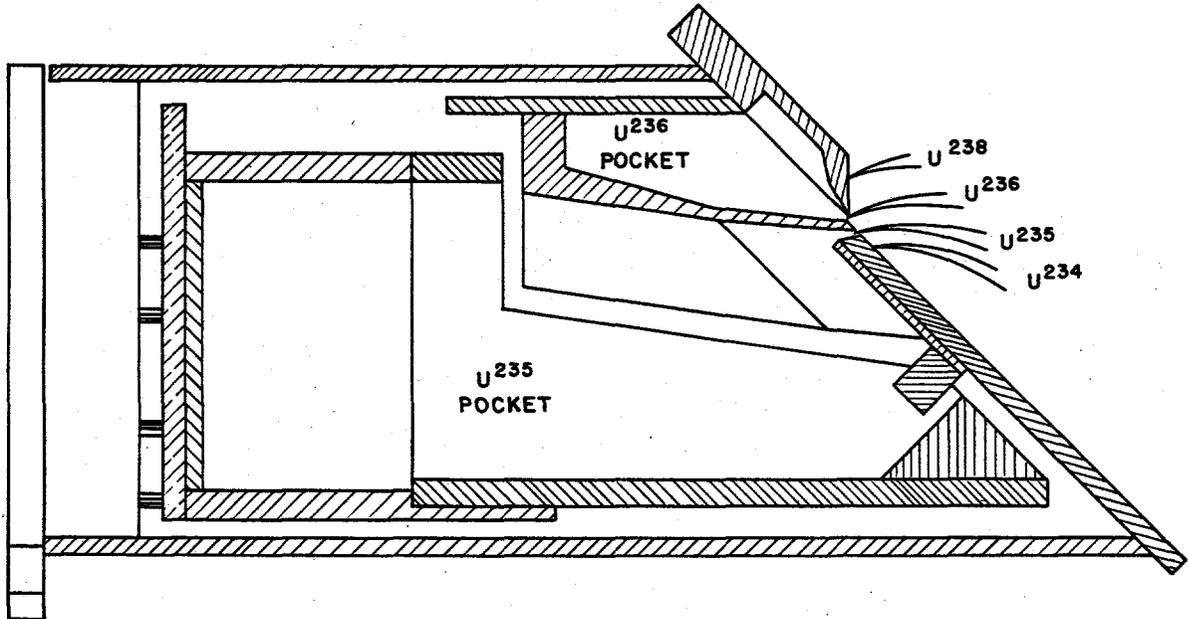


FIGURE II a. TWO - POCKET COLLECTOR

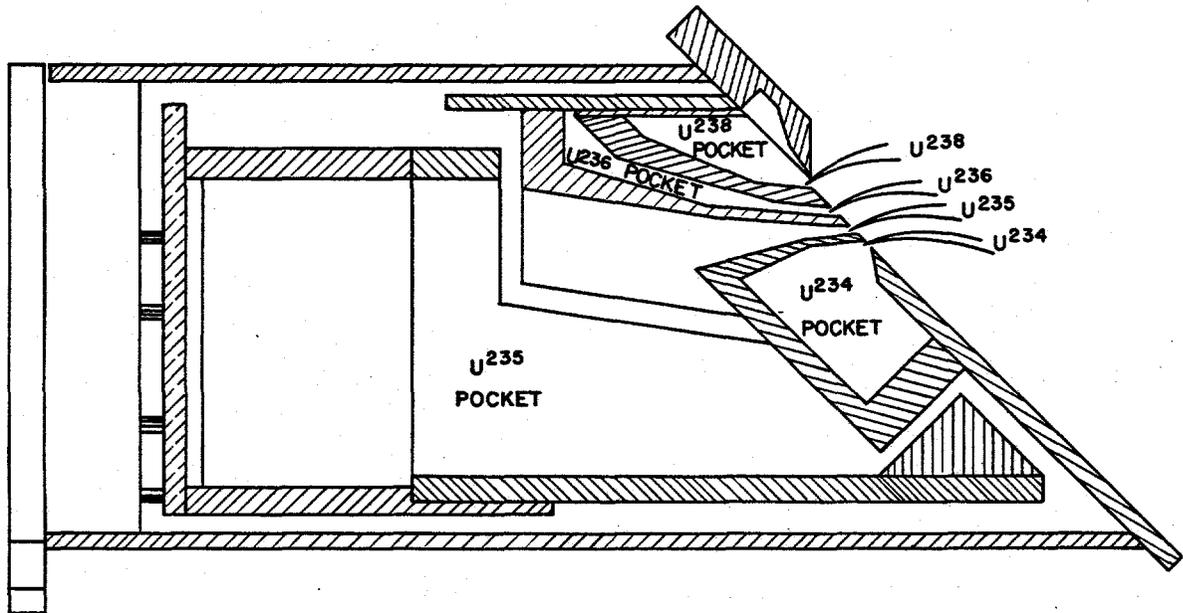


FIGURE II b. FOUR - POCKET COLLECTOR

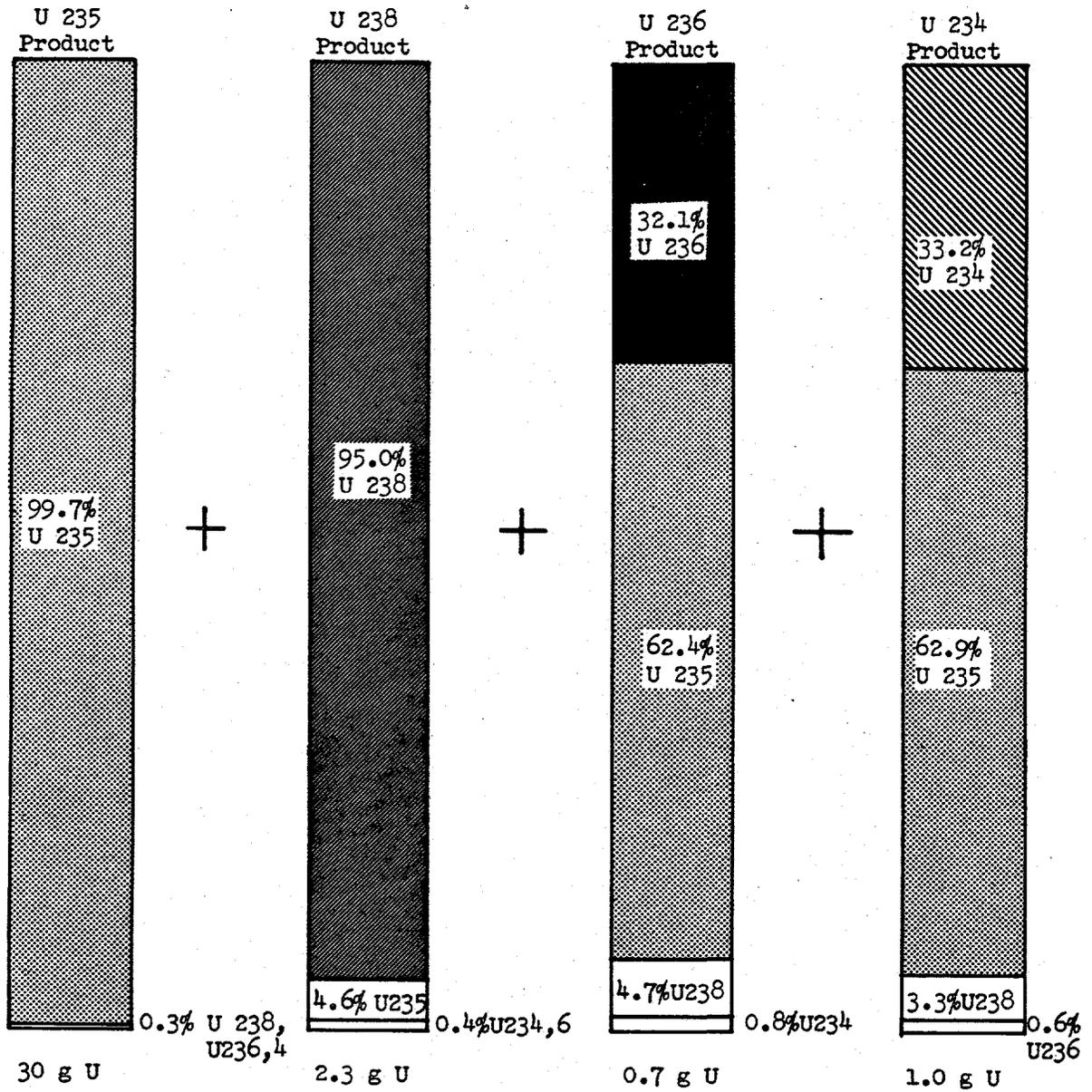


FIGURE 12. ISOTOPIC CONTENT OF PRODUCT PER RUN - STAGE I (FOUR-POCKET COLLECTOR)

SEPARATION AND ENRICHMENT OF SPENT REACTOR FUEL

It is predicted that spent fuel assemblies from the MTR reactor* will be depleted to 60% U 235 and enriched to 24% U 236. This uranium stock can be restored to 96% U 235 purity in one stage of mass-spectrographic enrichment. This section illustrates the isotope separation process at equilibrium as applied to processing of such MTR fuel assemblies. Performance parameters used are derived from the already completed U 236 separations as described in this report. The basic data assumed per separation run are:

Separation period	70 hrs
Down time	7 hrs
Working feed	450 gm U
Total product removal	39 gm
Make-up feed	41 gm
Recycling period	40 hrs
Total power consumption	9000 KWH

The table below lists the relative isotopic abundances of the separated fractions deposited in the collector for each run.

Principal Isotope	Isotopic Composition (wt. %)				Uranium (gm)
	U 235	U 236	U 238	U 234	
U 235	96.2	3.3	0.3	0.2	23.9
U 236	4.3	94.6	0.1	<0.1	8.4
U 238	1.5	2.5	95.9	0.1	5.5
U 234	24.8	11.5	5.2	58.5	1.6

Modifications in calutron geometry to permit increasing the volume of production are possible if purity requirements are relaxed.

The isotopic concentration of recycled uranium is changed with elapsed cycling due to non-uniform withdrawal of isotopes as product. As a result, the working feed bank at equilibrium differs isotopically from the MTR discard concentration. This trend and the consequent effect on product purity are charted in Figure 13.

* Goeller, H. E., The Cooling of Irradiated 25 MTR Fuel Assemblies and Its Effect on 25 Inventory in the MTR Project, ORNL-611, p. 8, March 15, 1950.

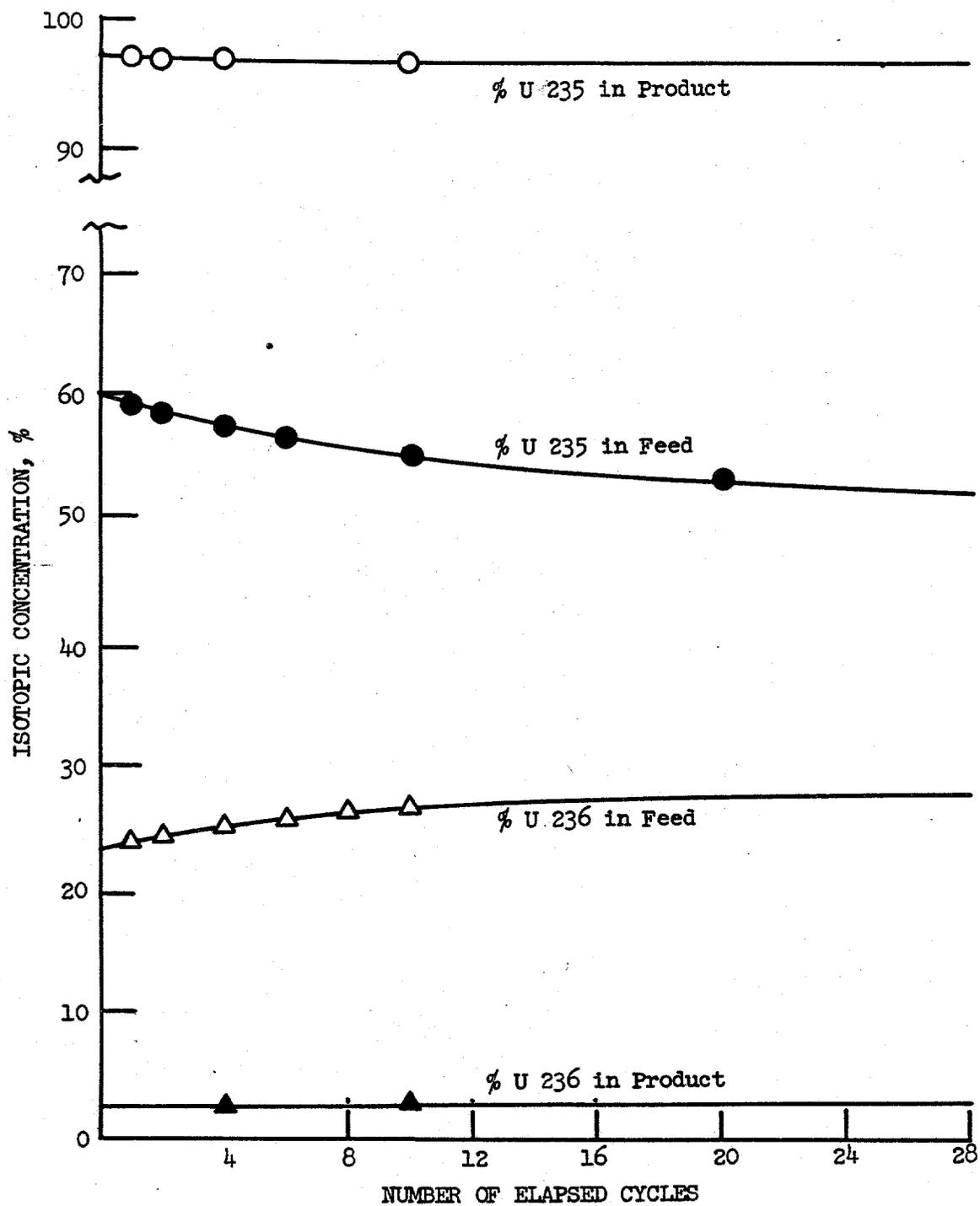


FIGURE 13. ISOTOPIC CONCENTRATION OF FEED AND U 235 PRODUCT AS A FUNCTION OF THE NUMBER OF ELAPSED CYCLES

ACKNOWLEDGEMENT

This project was accomplished through the joint effort of members of the Electromagnetic Research and the Y-12 Chemical Divisions. The following people in the Electromagnetic Research Division participated:

Mass spectrograph separation	M. Gillham L. Doss C. P. Shelton H. W. Ditchen
Calutron fabrication and assembly	C. C. Sharp J. D. Silver C. S. Patty E. T. O'Rourke
Calutron disassembly and wash	F. A. DiCarlo L. Childs
Recycle recovery operations	C. E. Harris F. A. DiCarlo V. O. Haynes N. K. Bernander R. D. Ellingson
Product recovery	C. C. Sharp

Members of the Y-12 Chemical Division participating include:

Mass analysis and sample recovery	R. F. Hibbs O. W. Briscoe W. D. Harman
Chemical and spectrochemical analysis	F. E. Clark A. Skritnansky J. E. Paterson
Uranium control and computing	F. C. Uffelman C. L. Pigott

Measurements of airborne alpha activity were made by the Health Physics Department under E. C. Struxness. B. Buckminster handled statistical computations and editorial assistance was provided by R. L. Murray.

APPENDIX A: PREVIOUS U 236 ENRICHMENT

Mass spectrographic separation of U 236 was initially done at Y-12 on a smaller scale in 1948 and 1949, yielding 12 mg of 22% U 236 product.* Since only 11 grams of feed material was available the standard Beta calutron was modified so as to reduce the vaporization rate and dispersion of uranium. The beam radius was reduced to 12 inches in order to confine unresolved deposits to a smaller area for more efficient recovery. To obtain adequate enrichment, U⁺ ions emergent from a 3" by 1/16" source slit were brought to a 1/64" collector slot with not greater than 4° half-angle of divergence.

The production goal was milligram amounts at intermediate U 236 purity. In the six elapsed cycles of the project, the following performance was achieved:

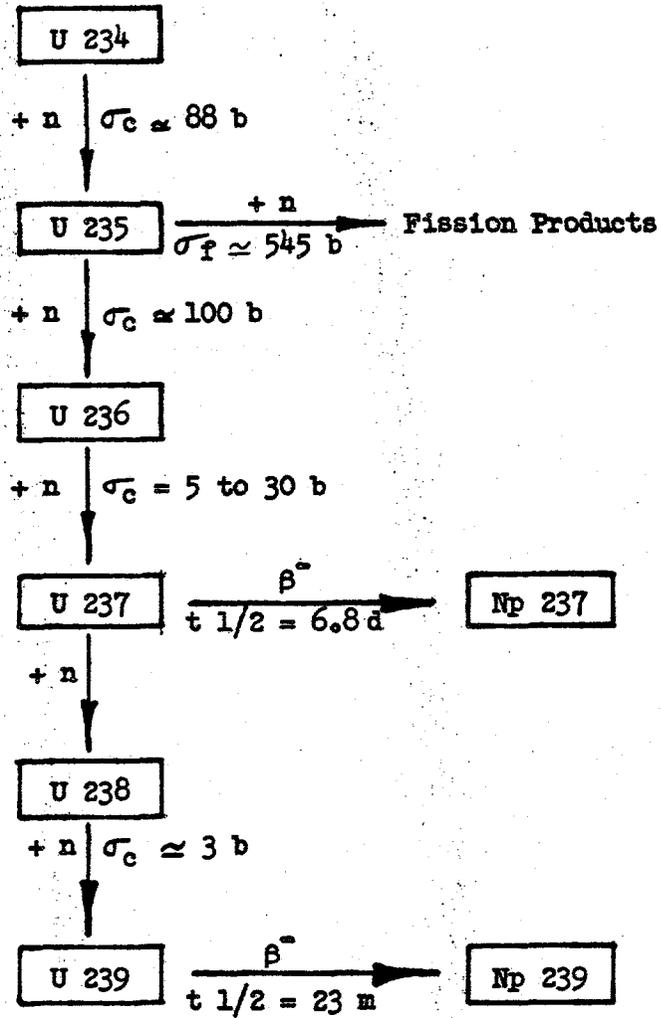
	Uranium (gm)	Isotopic Composition (%)		
		U 236	U 235	U 238
Feed: Starting	11.3	0.8	93.7	4.7
Throughput	50.2	0.8	92.9	5.5
Product: Run 1	0.0022	18	61	21
Run 2	0.0032	29	54	17
Run 3	0.0025	21	68	11
Run 4	0.0028	20	58	22
Run 5	0.0008	15	71	13
Run 6	0.0009	20	74	6
Recovery: Ending	8.8	0.7	91.8	6.7

The U⁺ currents averaged 0.5 ma and the vaporization rate 1 gm U/hr. The U 236 product enhancement relative to U 235 was 43 and the process efficiency approached 0.7%. Terminal recovery efficiency for U 236 was 93% of the six-cycle throughput or 70% of the starting supply.

* Savage, H. W., and Wilkinson, P. E., The Separation and Collection of U 236 by the Electromagnetic Process, Y-697, November 30, 1950.

APPENDIX B: ENRICHED IRRADIATED URANIUM FEED SUPPLY

<u>Batch</u>	<u>Wt. U</u>	<u>U 235</u>		<u>U 236</u>		<u>U 238</u>	<u>U 234</u>
	<u>(gm)</u>	<u>(gm)</u>	<u>(%)</u>	<u>(gm)</u>	<u>(%)</u>	<u>(%)</u>	<u>(%)</u>
1y	282.3	259.2	91.83	2.06	0.73	6.27	1.17
2y	299.1	276.2	92.33	2.27	0.76	5.73	1.18
3y	306.8	283.4	92.37	2.39	0.78	5.67	1.18
4y	290.8	267.0	91.83	3.37	1.16	5.81	1.20
5y	278.2	255.1	91.68	3.53	1.27	5.87	1.18
6y	303.2	277.9	91.67	3.91	1.29	5.85	1.19
7y	286.4	262.4	91.63	3.75	1.31	5.86	1.20
3p	64.6	59.4	91.92	0.70	1.09	5.81	1.18
8,9,10y	51.8	47.4	91.52	0.69	1.33	5.96	1.19
xwp	30.2	27.5	91.21	0.30	0.99	6.64	1.16
xz	1.4	1.3	91.62	0.02	1.20	5.98	1.20
xbx	8.5	7.8	91.81	0.06	0.72	6.68	0.81
Total	2203.3	2024.6	91.89	23.05	1.05	5.88	1.18

APPENDIX C: THERMAL NEUTRON REACTIONS WITH URANIUM ISOTOPES

APPENDIX D: U 236 PRODUCTION AND PROCESS PERFORMANCE, FIRST STAGE

Run	Production Uranium (gm)	Isotopic Composition (%)				Enhancement			Process Efficiency (%)
		U 236	U 235	U 238	U 234	236:235	236:238	236:234	
1	0.68	23.6	72.4	3.3	0.7	40	56	54	5.6
2	0.78	23.5	72.6	3.3	0.7	39	55	53	6.3
3	0.62	21.7	74.2	3.4	0.7	35	56	56	6.2
4	0.80	35.5	60.9	3.0	0.6	43	56	55	6.1
5	0.64	25.8	68.7	4.6	0.9	44	49	44	5.3
6	0.67	34.6	62.0	2.8	0.6	41	57	54	5.6
7	0.97	31.3	65.2	2.8	0.7	34	52	47	6.9
8	0.88	34.1	61.9	3.3	0.7	39	53	48	7.6
9	0.78	31.6	65.5	2.3	0.6	35	70	53	6.2
10	0.89	39.3	56.8	3.1	0.7	50	64	53	7.4
11	0.80	31.5	64.7	3.1	0.7	35	52	43	7.2
12	0.86	22.0	73.3	3.9	0.8	35	57	51	6.6
13	0.71	34.8	61.1	3.5	0.6	39	56	59	6.6
14	0.81	33.0	63.1	3.2	0.7	38	60	54	6.3
15	1.15	26.0	69.0	4.3	0.8	28	43	41	6.4
16	0.61	26.0	67.7	5.4	0.9	44	48	51	6.4
17	0.70	33.1	62.0	4.2	0.8	38	45	50	5.6
18	0.89	31.9	63.9	3.5	0.8	38	56	48	7.1
19	0.99	27.9	67.1	4.3	0.8	32	48	45	8.6
20	0.64	38.5	55.7	4.9	0.9	53	49	52	6.1
21	0.81	28.6	65.3	5.1	1.0	50	57	53	8.3
22	0.47	33.5	60.7	5.2	0.7	39	47	62	4.5
23	0.64	36.5	56.5	6.1	0.9	46	48	54	5.3
24	1.09	31.4	62.0	5.7	0.9	40	50	56	6.9
25	0.75	33.1	60.8	5.2	0.9	52	49	51	5.3
26	0.86	31.8	61.2	0.1	1.0	37	41	42	5.8
27	0.57	33.8	58.7	6.5	1.0	46	57	58	5.6
28	0.84	33.8	60.1	5.2	1.0	40	58	55	6.5
29	0.75	32.9	60.7	5.4	0.9	39	57	55	6.0
30	0.58	41.3	51.1	7.0	0.6	62	67	123	7.2
31	0.73	35.8	58.0	5.1	1.1	42	66	52	6.4
32	0.78	41.2	51.9	6.1	0.9	53	66	73	7.4
33	0.72	36.0	55.8	7.1	1.1	49	60	61	8.0
34	0.39	33.1	60.6	5.2	1.1	38	60	50	6.6
35	0.60	36.8	56.0	6.2	1.1	44	58	55	5.6
36	0.80	35.2	56.8	6.8	1.3	44	56	51	6.1
37	0.78	36.9	55.3	6.6	1.2	45	50	50	5.8
Total	28.04	32.1	62.4	4.7	0.8	40	54	52	6.2

APPENDIX E: U 235 PRODUCTION AND PROCESS PERFORMANCE, FIRST STAGE

Run	Production Uranium (gm)	Isotopic Composition (%)				Enhancement			Process Efficiency (%)
		U 235	U 236	U 238	U 234	235:236	235:238	235:234	
1	35.9	99.8	0.06	0.10	0.02	14	64	64	10.2
2	39.4	99.8	0.06	0.09	0.04	14	71	32	11.2
3	26.2	99.8	0.06	0.10	0.04	14	74	35	10.2
4	34.4	99.8	0.07	0.09	0.03	19	71	43	10.0
5	35.6	99.8	0.08	0.13	0.01	11	57	138	9.8
6	30.3	99.9	0.07	0.07	0.01	20	91	130	10.1
7	35.6	99.8	0.07	0.13	0.05	20	49	26	11.3
8	33.0	99.7	0.08	0.11	0.07	18	66	20	11.8
9	30.2	99.8	0.06	0.09	0.04	23	79	35	10.6
10	36.8	99.7	0.12	0.14	0.04	12	50	34	10.9
11	25.6	99.8	0.07	0.09	0.04	20	80	35	10.3
12	33.3	99.8	0.05	0.08	0.05	17	109	31	10.1
13	25.5	99.8	0.07	0.12	0.04	21	73	31	9.9
14	28.3	99.7	0.11	0.12	0.05	13	67	30	9.1
15	33.4	99.7	0.11	0.14	0.08	11	67	21	9.5
16	27.6	99.7	0.08	0.14	0.08	13	62	19	9.8
17	29.6	99.7	0.09	0.16	0.08	15	51	19	9.5
18	34.0	99.7	0.08	0.17	0.05	17	48	30	11.2
19	33.1	99.7	0.09	0.13	0.07	14	72	23	8.9
20	29.6	99.7	0.09	0.16	0.04	15	51	39	9.5
21	37.0	99.7	0.06	0.16	0.06	14	54	26	11.6
22	19.5	99.7	0.10	0.15	0.05	14	67	36	7.8
23	29.3	99.7	0.10	0.14	0.05	14	80	38	9.4
24	33.6	99.7	0.09	0.15	0.06	14	77	34	8.5
25	29.7	99.7	0.08	0.20	0.06	17	54	32	8.7
26	32.3	99.6	0.14	0.16	0.08	10	69	23	9.7
27	23.7	99.7	0.07	0.15	0.08	18	90	27	8.6
28	31.2	99.7	0.08	0.15	0.07	18	82	31	10.0
29	27.1	99.7	0.09	0.15	0.09	16	87	24	9.1
30	25.4	99.7	0.07	0.14	0.07	19	106	34	9.8
31	27.8	99.7	0.08	0.13	0.06	18	105	40	9.9
32	27.2	99.7	0.09	0.13	0.06	17	112	39	9.3
33	28.6	99.7	0.09	0.18	0.07	14	87	37	11.7
34	28.2	99.7	0.07	0.18	0.07	21	76	34	10.4
35	22.5	99.7	0.08	0.16	0.07	19	91	34	8.6
36	30.7	99.7	0.09	0.17	0.07	15	88	36	9.2
37	33.9	99.6	0.09	0.22	0.07	16	65	34	9.9
Total	1125.0	99.7	0.08	0.14	0.06	16	82	35	9.9

APPENDIX F: U 235 and U 236 PRODUCTION AND PROCESS PERFORMANCE, SECOND STAGE

U 236 Yield

Run	Production	Isotopic Composition (%)				Enhancement			Process
	Uranium (gm)	U 236	U 235	U 238	U 234	236:235	236:238	236:234	Efficiency (%)
1	0.34	96.7	3.1	0.2	0.05	61	68	52	4.8
2	0.39	95.3	4.2	0.4	0.15	45	54	19	5.8
3	0.27	94.9	4.6	0.4	0.07	41	57	44	4.2
4	0.27	94.8	4.6	0.5	0.07	42	59	48	4.5
5	0.22	95.5	4.0	0.4	0.06	48	75	56	5.3
6	<u>0.27</u>	95.2	4.1	0.6	0.09	47	62	40	5.6
Total	1.76	95.4	4.1	0.4	0.09	47	61	36	5.0

U 235 Yield

Run	Production	Isotopic Composition (%)				Enhancement			Process
	Uranium (gm)	U 235	U 236	U 238	U 234	235:236	235:238	235:234	Efficiency (%)
1	1.09	98.2	1.7	0.07	0.04	29	106	34	8.1
2	1.02	98.5	1.3	0.13	0.06	38	78	24	8.0
3	0.84	96.7	3.0	0.29	0.08	17	40	20	6.7
4	0.84	97.7	2.0	0.29	0.06	25	54	27	7.8
5	0.72	97.6	2.1	0.25	0.05	23	68	35	8.8
6	<u>0.69</u>	98.4	1.5	0.12	0.04	34	160	46	7.4
Total	5.20	97.9	1.9	0.18	0.06	26	71	26	7.6

APPENDIX G: ENHANCEMENT THEORY AND COMPUTATION

Definitions of enhancement (N) are given for the separated isotope U 236 with derivation of the relationship between enhancement and isotopic concentration. The analysis is carried out for the two-stage electromagnetic separation. Independence of the enhancement factor relative to feed concentration is established.

Assume the following notation:

<u>Subject Material</u>	<u>Isotopic Composition</u>			
	<u>% U 236</u>	<u>% U 234</u>	<u>% U 235</u>	<u>% U 238</u>
Stage I Feed	v_o	w_o	x_o	y_o
Stage I Product	v_p	w_p	x_p	y_p
Stage II Product	v'_p	w'_p	x'_p	y'_p

Formulas for single and multiple-stage enhancement of U 236 relative to U 235 are:

$$\begin{array}{ccc}
 \text{Stage I (N}_{vx}) & \text{Stage II (N}'_{vx}) & \text{Overall (N}_{vx}N'_{vx}) \\
 \frac{v_p/x_p}{v_o/x_o} & \frac{v'_p/x'_p}{v_p/x_p} & \frac{v'_p/x'_p}{v_o/x_o} \quad (1)
 \end{array}$$

Analogous expressions define U 236 enhancement relative to mass 234 and 238 fractions. Isotopic concentrations may be expressed as either weight or atom percent for calculating enhancement.

Formulas for isotopic concentration of U 236 product are:

$$\begin{array}{cc}
 \text{Stage I} & \text{Stage II} \\
 v_p/v_p + w_p + x_p + y_p & v'_p/v'_p + w'_p + x'_p + y'_p \quad (2)
 \end{array}$$

Substituting (1) in (2) and simplifying gives:

$$\begin{array}{ccc}
 \text{Stage I} & & \text{Stage II} \\
 v_0/v_0 + \frac{w_0}{N_{vw}} + \frac{x_0}{N_{vx}} + \frac{y_0}{N_{vy}} & & v_0/v_0 + \frac{w_0}{N_{vw}N'_{vw}} + \frac{x_0}{N_{vx}N'_{vx}} + \frac{y_0}{N_{vy}N'_{vy}} \quad (3)
 \end{array}$$

Product U 236 concentration as a function of enhancement is illustrated in Figures 14 and 15. The range of enhancement performance for each stage is indicated.

The interrelation between enhancement and isotopic abundance of feed remains to be determined. In the collector box, similar U 235 and U 236 slots are displaced one mass unit, center to center. The relative intensities of isotopic ion beams received through these slots are proportional to the atom concentrations in the feed. The ratio of isotopic yields from a U 236 pocket (V) and adjacent U 235 pocket (R) is given by:

$$\frac{v_p \text{ in V}}{x_p \text{ in R}} = \frac{v_0}{x_0} \cdot \frac{\text{V retention efficiency}}{\text{R retention efficiency}}$$

From (1), the functional relationship between v_p in V and x_p in V is obtained:

$$\frac{v_p \text{ in V}}{x_p \text{ in V}} = N_{vx} \cdot \frac{v_0}{x_0}$$

Substituting for v_p in V in (4):

$$N_{vx} = \frac{x_p \text{ in R}}{x_p \text{ in V}} \cdot \frac{\text{V retention efficiency}}{\text{R retention efficiency}}$$

In the same manner, other enhancement factors can be shown to be independent of isotope concentration in the feed.

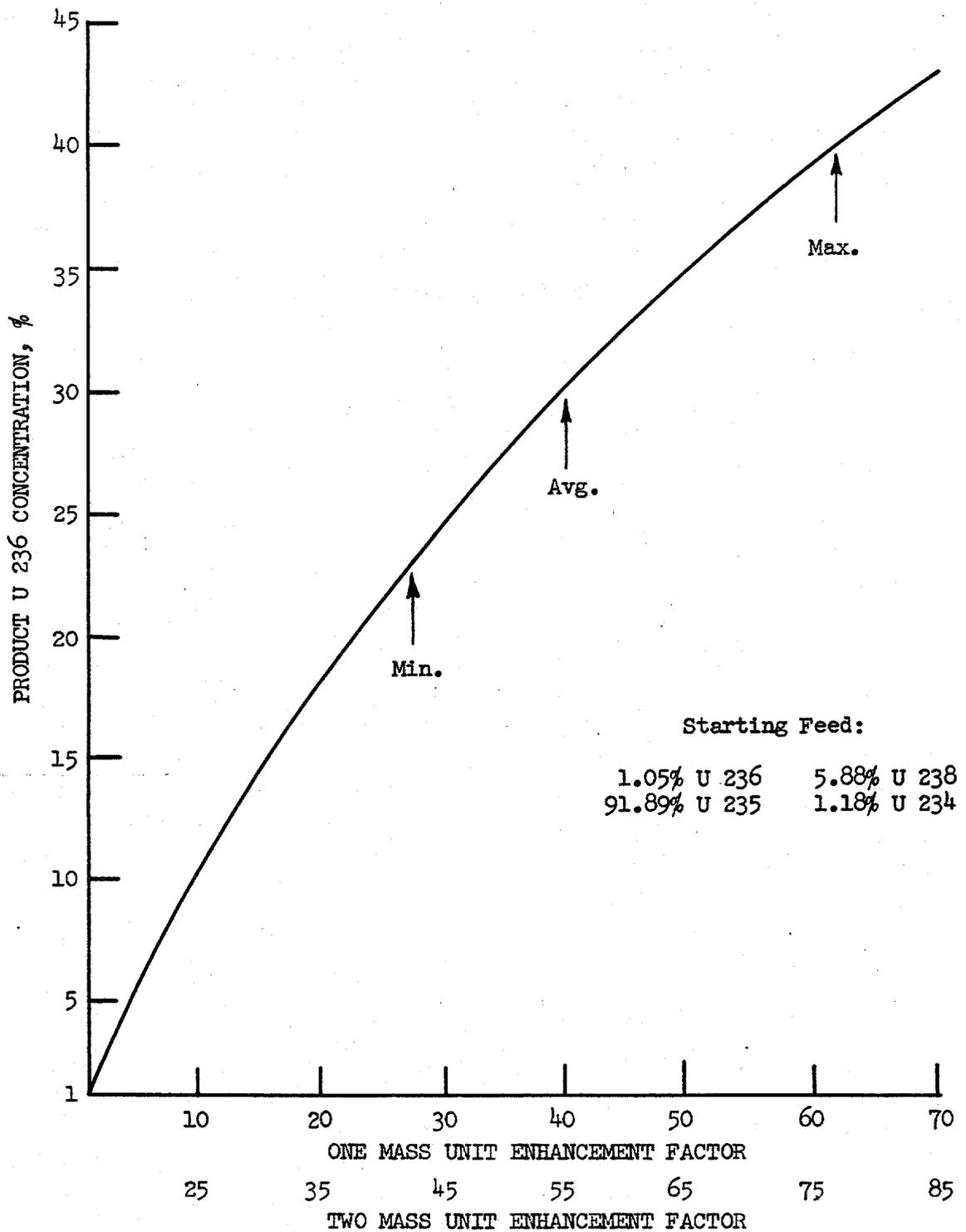


FIGURE 14. VARIATION OF FIRST STAGE PRODUCT U 236 CONCENTRATION ON APPLYING VARIOUS ENHANCEMENT FACTORS TO STARTING FEED

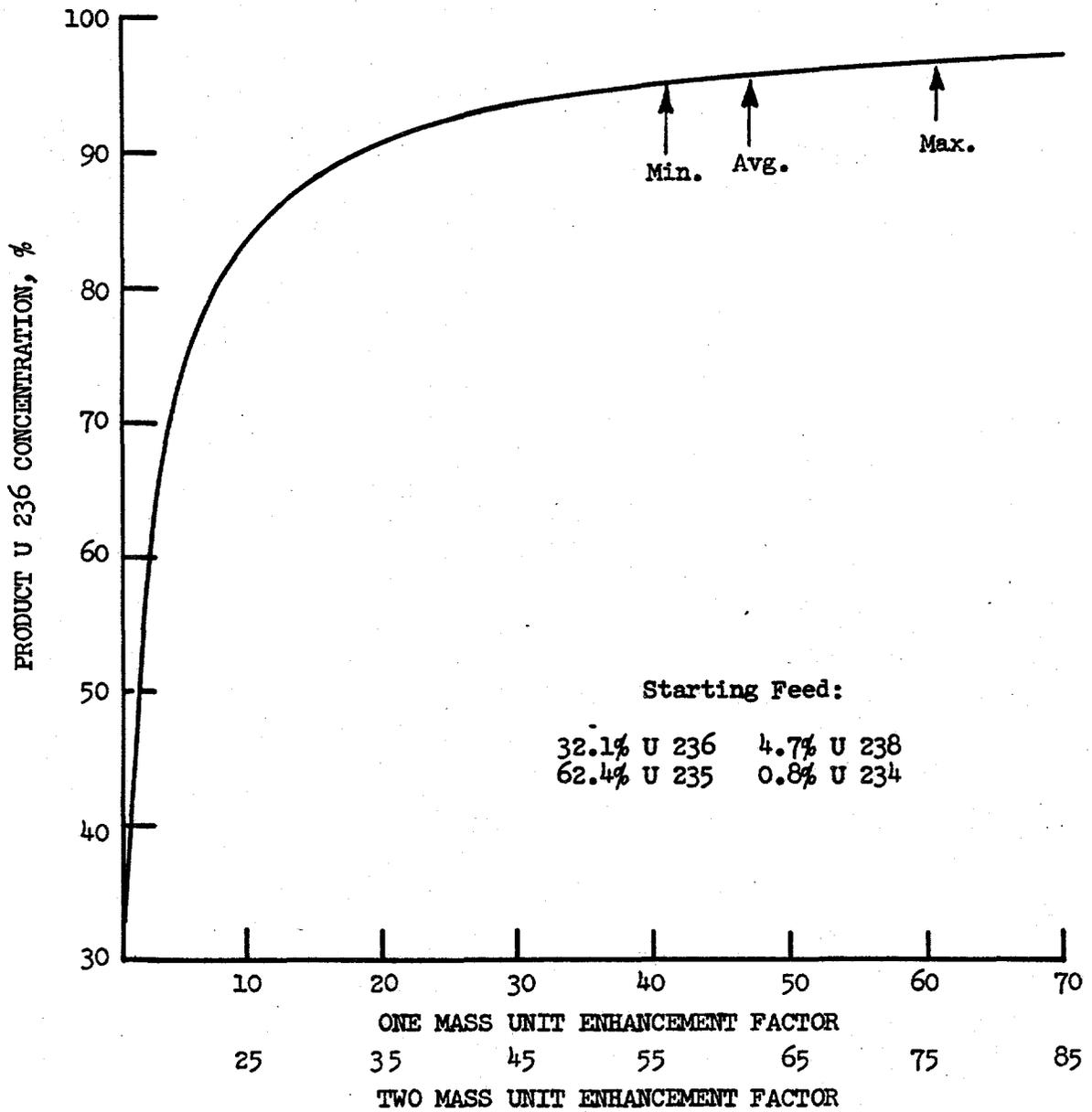


FIGURE 15. VARIATION OF SECOND STAGE PRODUCT U 236 CONCENTRATION ON APPLYING ENHANCEMENT FACTORS TO STARTING FEED

APPENDIX H: LIMITS OF ERROR IN MASS ANALYSES

The Y-12 Spectrometer Laboratory measured the isotopic constituents of U 236 product using UF_6 gas in a 60-degree Nier-type spectrometer. Two series of measurements were made, each consisting of four determinations, with a clean ion source and sample system. If results were reproducible within the usual limit of error, the average value was reported, otherwise the sample was re-analyzed. Possible inaccuracies in the assays due to spectrometer bias were not included. Limits of error representing a 95% confidence interval, based on analyses of standard samples, are given below:

<u>Isotope</u>	<u>Relative Isotopic Abundance, wt. %</u>	
	<u>Product Batch 2V-1</u>	<u>Product Batch 1V-0</u>
U 236	96.65 ± 0.07	42.38 ± 0.20
U 235	3.09 ± 0.03	53.80 ± 0.20
U 238	0.21 ± 0.02	3.16 ± 0.03
U 234	0.05 ± 0.01	0.66 ± 0.05

APPENDIX I: SPECTROGRAPHIC ANALYSIS OF U 236 PRODUCT OXIDE

A sample of U 236 product oxide (Batch 2V-2) delivered to the Y-12 Spectrographic Laboratory was analyzed for trace contaminants in a 21-foot Jarrell-Ash, 15,000 line per inch grating spectrograph. The total impurity is a maximum of 2500 parts per million by weight in U_3O_8 , corresponding to a chemical purity of 99.75%. Two values were obtained for some elements because the sample could not be ground prior to analysis, thus making some variations in the aliquots. This analysis is probably representative of other U 236 product oxides. Contaminating elements in parts per million, by weight in U_3O_8 , are:

Ag	<1	Mg	10, 1
Al	<20, 60	Mn	12
B	25	Mo	300, <100
Ba	25	Na	125, 30
Be	<0.2	Ni	60, 500
Bi	<10	P	100
Ca	30	Pb	70
Cd	<10	Si	600
Co	<20	Sn	<40
Cr	30	Sr	<100
Cu	6	Ti	<100
Fe	200	V	<100
K	<100	Zn	<40
Li	<10		

APPENDIX J: MEASURED PROPERTIES OF URANIUM 236

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Half life	2.46×10^7 years	Jaffey, Diamond, Hirsch, Mech, <u>Phys Rev</u> <u>84</u> , 785 (1951)
Alpha particle energy	4.499 ± 0.004 Mev	Ibid.
Spontaneous fission	7.7 ± 4.4 fissions/g-hr	Private communication, W. M. Manning to R. S. Livingston, June 16, 1950
Isotope shift of U 236 line spectrum	4244.226 A	Smith, Stukenbroeker, McNally, AEC-3218 (1949)
Pile capture cross section from yield of Pu 238	23.5 ± 4.7 barns	P. R. Fields, G. L. Pyle, ANL-4490 (1950)
Thermal-neutron capture cross section	5.8 ± 1.8 barns	H. Pomerance, ORNL-1216 (1951)
Average fission cross section in the fast reactor neutron spectrum	0.181 ± 0.0135 barns	E. Journey, LA-1250 (1951)
Fission cross section of U 236 as a function of energy	0.69 Mev - 0.04 barns 1.00 Mev - 0.36 barns 2.55 Mev - 0.53 barns 14.1 Mev - 1.65 barns	W. Nyer, LA-1258 (1951)

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