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FINAL REPORT
PRODUCTION TEST IP-2-A
ENRICHED URANIUM CONVERSION AND STABILITY
TEST

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FINAL REPORT PRODUCTION TEST IP-2-A ENRICHED URANIUM CONVERSION AND STABILITY TEST

INTRODUCTION

Production Test IP-2-A⁽¹⁾ was undertaken as part of the HAPO enriched uranium testing program.⁽²⁾ The purpose of the test was 1) to determine the conversion ratio of alternated enriched uranium fuel segments and lithium aluminum target slugs, and 2) to determine the stability of solid and cored enriched uranium with this type of load.

The test consisted of an irradiation of two twelve-tube pile charges, each of them forming a sixteen tube square without corners. The first twelve tubes were a "striped" load containing solid fuel elements enriched to .95 percent U-235 alternated with lithium-aluminum target slugs. The fuel elements of the second array were 1/2" cored, but canned with the core plugged to give the same O.D. and external appearance as a solid element. To determine fuel element stability, the irradiation was essentially a run-to-rupture test. The central four-tubes of each square were designated for special extraction after the irradiation to determine conversion ratio.

(1) HW-46099, "Production Test IP-2-A, Enriched Uranium Conversion Ratio and Stability Test," L.W. Lang, 11-12-56.

(2) HW-40555, "Enriched Uranium Testing Program," L.W. Lang, 1-11-56.

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SUMMARY

The cored tubes were discharged with 137 days of in-pile residence after a failure occurred in one of the charges at a calculated exposure of 841 MWD/T. Five days later the tubes in the solid array were discharged after a failure was sustained at 755 MWD/T.

A summary of the extraction data, given in Table No. 1, shows the conversion ratios from this type of loading are four percent higher on a atom-basis than from natural U loadings. Final conversions at 800 MWD/T as shown in Table No. 2 are roughly twelve percent higher than natural U loadings due to the higher initial ratios and the reduced rate of product burn-out.

CONCLUSIONS

Neither solid nor cored enriched uranium of metal quality comparable to that used in this test has adequate stability to reach a routine goal exposure of more than 500 MWD/T at the specific powers of the test, 70-80 KW/foot. From post-irradiation measurements of diameter growth of the cored slugs, it might be concluded that many of the slugs were split, but not sufficiently to rupture the aluminum jacket.

The conversion ratios are near those that have been used in process studies; see Table No. 3. Although uncertainties are introduced in exposure measurements, and column reactivity, the conversion ratios have an estimated reliability of 2 percent.

DISCUSSION

Description of the Test

The tube pattern for the test and metal for individual tube make-up is shown in Table No. 4. The reason for charging the tubes in a square was to eliminate as much as possible the effect of natural U columns on the inner four tubes of each array. It was reasoned that the inner tubes would be in an environment closer to that that would prevail in a full pile charging. If the power of inner and outer tubes were the same, it would be indicative that the reactivity of the load was nearly correct. Columns were shortened from natural U length so as to maintain tube powers near that of natural U.

The placement of the N slugs was changed within each individual load; note A and B tube make-up; in order to minimize front to rear flux peculiarities that could affect the conversion ratio split.

Panelit readings and outlet temperatures were taken each shift to calculate tube powers and exposure. Pre and Post-Irradiation measurements were taken of all test slugs.

Upon discharge, the central four tubes of each array were designated for special extraction; the N-slugs were sent to Savannah River and the E-metal extraction was performed by individual tube loadings in the Redox Plant.

A schedule of discharge was designated to be contingent upon a rupture occurring in the special metal or the metal achieving a relatively high exposure.

TABLE NO. 1

INITIAL AND FINAL CONVERSION RATIOS
(Tritium Expressed as Pu g atom equivalents)

	Initial g/MWD			706 MWD/T			833 MWD/T			Percent Increase	
	<u>Pu</u>	<u>Trit.</u>	<u>Total</u>	<u>Pu</u>	<u>T₂</u>	<u>Total</u>	<u>Pu</u>	<u>T₂</u>	<u>Total</u>	<u>Initial</u>	<u>Final</u>
Nat. U, Solid	.974	-	.974	.850		.850					
Nat. U, Cored	.936	-	.936				.796	-	.796		
Nat. U, I & E	1.004	-	1.004								
.94 Enriched Solid Li-Al.	.812	.197	1.009	.743	.197*	.940				3.6	10.6
.94 Enriched Cored - Li-Al.	.770	.203	.973				.695	.203*	.899	4.0	12.9

* No allowance for tritium decay.

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TABLE NO. 2 ⁽⁷⁾
EXTRACTION DATA AND CALCULATED CONVERSIONS

Tube No.	Measured Exposure	Pu(1,2) (g/Ton)	Tritium (cc STD-- (5) Theoretical)	Tritium (Actual plus He ³)	Pu	Yield/MWD T ₂ (g Equiv)	Total
<u>Solid Elements</u>							
2062	682.3	522	556	537 + 25.2	.765	.202	
2162	703.3	528	562	548 + 41.2	.751	.198	
2163	731.5	522	569	551 + 38.7	.714	.193	
2063	736.2	<u>(3)</u>	578	559 + 37.2	—	<u>.195</u>	—
Avg.		526			<u>.743</u>	.197	.940
<u>Cored Elements</u>							
2068	834.2	579	588	578 + 33.3	.695	.202	
2069	822.1		605	570 + 42.0		.212	
2168	828.8		589	586 + 31.1		.204	
2169	<u>846.6</u>	—	<u>576</u>	563 + 33.3	—	<u>.194</u>	—
Avg.	833.0	579 ⁽⁴⁾			.695	.203	.899

- (1) Wt. U in Solid Tube .08595 Tons
 (2) Wt. U in Cored Tube .07425 Tons
 (3) Product mixed from Tube 2063
 (4) Pu combined for four cored tubes
 (5) Based on measured amounts of He⁴, He³
 (6) Tritium based on Pu g equivalents
 (7) Analytical Data as Reported in Appendix.

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TABLE NO. 3

MEASURED AND CALCULATED E-N VALUES FOR OLD FILES

	<u>Pu g/MWD</u>	<u>Tritium g atom Equiv/MWD</u>	<u>Total g atom Conversion</u>
Nat. U, Old Pile	.974	--	.974
E-N, Reference 3	.794	.188	.982
Reference 4	.776	.194	.970
Reference 5	.806	.199	1.005
E-N, This Test	<u>.812</u>	<u>.197</u>	<u>1.009</u>
E-N, If I&E Geometry	.842	.182	1.024-
E-N, If I&E, with Lithium Enrichment	.842	.197	1.039 ←
E-N, If I&E with Lithium Enrichment and Fringe Poison	.842	.212	1.054

(3) HW-39678, "Analysis of Enriched Uranium Production of Tritium vs Conventional Alloy Loading," L. W. Lang, 10-26-55.

(4) XX-1582, 4-27-56.

(5) CLIV-31, 8-14-57.

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TABLE NO. 4

CHARGING PATTERN FOR TEST MATERIAL

Row/Column	60	61	62	63	64	65	66	67	68	69	70	71
23	U	U	U	U	U	U	U	U	U	U	U	U
22	U	U	A	B	U	U	U	U	C	D	U	U
21	U	A	B	A	B	U	U	C	D	C	D	U
20	U	B	A	B	A	U	U	D	C	D	C	U
19	U	U	B	A	U	U	U	U	D	C	U	U
18	U	U	U	U	U	U	U	U	U	U	U	U

Tubes were charged in two four-by-four squares as shown above.

INDIVIDUAL TUBE MAKE-UP

<u>U</u> (48 tubes)	<u>A</u> (6 tubes)	<u>B</u> (6 tubes)	<u>C</u> (6 tubes)	<u>D</u> (6 tubes)
Reg. Dummy Pattern	11-8" Perfs. 7-8" SA	11-8" Perfs. 7-8" SA	11-8" 7-8" SA	11-8 7-8" SA
30 - 8" Nat. U (Slugs in corner charges individually identified)	2 - E Solid 1 - N 6 - E Solid 1 - N 6 - E Solid 1 - N 6 - E 1 - N 6 - E 1 - N 4 - E	4 - E Solid 1 - N 6 - E Solid 1 - N 6 - E Solid 1 - N 6 - E 1 - N 6 - E 1 - N 2 - E	2 - E Cored 1 - N 6 - E Cored 1 - N 7 - E Cored 1 - N 6 - E 1 - N 6 - E 1 - N 4 - E	4 - E Cored 1 - N 6 - E Cored 1 - N 7 - E 1 - N 6 - E 1 - N 6 - E 1 - N 2 - E

E - Cored 1/2" Cored, 6-inch slugs .94 percent U-235

E - Solid 6-inch slugs .94 percent U-235

N - Std. Lithium-aluminum alloy pieces - 4" long ~ HW 46099

U - 8" - natural uranium

The central four tubes in each array had a nominal goal exposure of 62.5 MWD/tube. A special pick-up was required for the eight central tubes after irradiation, and tube identities were maintained.

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Data Obtained

In each array three sets of power readings were averaged from individual tube readings:

- 1) The Inner Four E-N Tubes.
- 2) The Outer Eight E-N Tubes.
- 3) The Uranium Tubes at the four corners of the 16-Tube array.

By comparing the ratio of the averages, information was hoped to be obtained on the reactivity matching of the E-N with natural uranium columns and of the effect of exposure on the reactivity of the columns. These data were quite erratic presumably due to asymmetrical rod movements on either side of the load and uncertainties introduced by the measurements themselves. Trend data, however, is plotted for each array for the ratios in Figure 1. The interpretation of this data is considered later in the report.

A rupture was sustained in the solid uranium at 755 MWD/T and the central tubes were discharged at 682, 703, 732, and 736 MWD/T exposures. Previously, a cored failure had occurred at 841 MWD/T. The central tubes of the cored array had achieved exposures of 834, 822, 829 and 847 MWD/T. Although the indicated exposures are less for the solid slugs, their irradiation time was slightly longer -- the actual weight of the uranium being more per column with the solid slugs.

Warp and diameter were determined for all of the E-N columns. The averages of Table No. 5 were obtained.

The tritium extraction data is contained in the Appendix and summarized in Table No. 1. Tritium and two isotopes of helium are contained in the irradiated lithium-aluminum slugs. An atom of He^4 is formed for each atom of tritium by Li^6 capture of a neutron. The He^3 is the decay product of tritium, which has an approximate 12 year half-life. Volumetric measurement of helium with an isotopic analysis can be compared with extracted tritium to double check yields. Where these yields are not in agreement, the helium measurement is felt to be the best reference of actual tritium formed. Thus the theoretical column of Table No. 1 and appendix data is based on helium measurements. The actual tritium is also shown. To rationalize the theoretical tritium with the measured value, one-half the volume of He^3 must be added.

Due to the high cost of individual tube extraction for Pu, it was decided to analyze the four cored tubes in one dissolution, rather than individually as done for the solid elements. Tube No. 2063 was not individually analyzed due to contamination with uranium and Pu from another source. The data as obtained from K. M. Harmon is given in the Appendix and is summarized in Table No. 1.

Interpretation of Test Results

Since the irradiation of this test (and partially due to this test) the use of solid and cored enrichment elements have been largely terminated in favor of internally and externally cooled enriched elements. It had become apparent before the analyses that coring was not an adequate solution to either normal

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BM-5300-055 (6-58) AEC-GE RICHMOND, WASH.

FIGURE NO. 1

PLOT OF RATIO OF INNER E-N
TUBE POWER TO OUTER VS TIME

Indicates

Outer > Inner Reactivity Low

Outer = Inner Reactivity Balanced

Outer < Inner Reactivity High

CHARGE PATTERN

TABLE NO. 5

DIMENSIONAL STABILITY* OF TEST E-METAL

	Diameter		Warp	
	<u>Solid</u>	<u>Cored</u>	<u>Solid</u>	<u>Cored</u>
Central 4 Tubes	-7.3 mil	+14.6	+ 13 mil	+ 26.6
Outer 8 Tubes	-4.0 mil	+20.8	+ 16	27.3
Nat. Controls	-5.7 mil		+ 12	

* Statistical analysis of the data concluded that the differences in diameter between the inner and outer tubes were significant for both cored and solid slugs and that the warp for the solid slugs was significantly greater.

or enriched slug stability problems. Thus the cored data from much of the test was downgraded inasmuch as its applicability is not as great as data from the solid slug irradiations.

Fuel Element Stability

The stability testing feature of the irradiation was essentially a run-to-rupture test of the two twelve-tube arrays. It was intended to give an indication of the comparative performance between the cored and solid E-metal, as well as the standard natural uranium elements which were solid geometry at the time of testing.

The ruptures occurred at relatively low goal exposure for both the solid and cored slugs, a calculated 755 MWD/T and 841 MWD/T, respectively. The average piece exposure at failure was 2.01 MWD for the cored elements and 2.24 MWD for the solid. The slug power averaged 53.0 KW/ft with a maximum power near 70.0 KW/ft. Due to the larger number of tubes that would be involved in a full-pile load, ruptures would be anticipated at lower exposures than the discharge exposure for the test material. Also, the outlet temperatures for these tubes was slightly lower than natural uranium charges. Both failures were attributed to core splitting.

The dimensional data is noteworthy from two standpoints:

- 1) The greater warp experienced in the outer tubes of both arrays.
- 2) The diameter growth and warp of the cored slugs.

The diameter growth of the cored slugs in light of subsequent examination of irradiated cored fuel elements was probably due to splitting of the core. The cored slugs were later shown to be prone to core splitting and ultimate failure by this mechanism. Apparently this type of splitting is initiated at the core with subsequent cycling extending the crack to the outer diameter. It can occur without immediate jacket failure, although accelerating ultimate failure by jacket penetration. The pile was cycled more than normal during the irradiation since new coolant pumps had just been installed, and were being production tested.

The observation of greater warp in the outer charges might be due to greater asymmetry of flux experienced by the metal in these tubes.

It would be concluded from the stability tests that neither cored or solid E-N charges are sufficiently stable to routinely irradiate at present powers and exposures.

Reactivity

The reactivity of these columns is important in determining the tritium portion of the conversion ratio. If the reactivity is low, too much lithium in the columns, the tubes are importing neutrons and will give high conversions for tritium production. If the reactivity is high, too few lithium slugs, the columns will export neutrons and the tritium conversion will be low. To determine the true reactivity requirement in a test such as this is difficult in that power requirements must also be met. The power of a full E-N column

in a natural U flux is roughly twelve percent higher than a column of green natural uranium fuel elements. Thus the column length had to be shortened by several slugs at each end of the column. These slugs were replaced with aluminum dummies downstream and a water column was allowed upstream. Thus the reactivity of the columns was less than had full column charging been possible (with the same uranium-lithium ratio).

Were it not necessary to "short-charge," the columns, the manner of loading "inner" and "outer" columns would be effective in demonstrating the E-N columns were reactivity matched with natural U tubes. If the reactivity of the columns was less than natural U tubes, the outer E-N charges would have greater power than the center tubes. If reactivity were greater, the inner tubes would have higher power than the outer tubes, due to the fact that they would see the increased flux from the outer tubes.

In actuality, as shown in Figure No. 1 for the solid load, the reactivity of the columns are slightly low. If full columns had been charged, it would have increased the reactivity somewhat. Thus it might be concluded that the reactivity is about right for the cored slugs but slightly on the low side for the solid elements: The cored array had one extra fuel element per tube. The indicated four percent difference in conversion between the cored and the solid arrays would be less were the reactivity of the two columns equivalent.

The reactivity of the columns appears to decrease with time as compared to the natural U columns. This is primarily due to reactivity build-up in the adjacent columns of natural U and agrees well with theoretical calculations. The reactivity of the enriched uranium columns remains relatively constant over the irradiation period of the test. The apparent increase in reactivity shown near the test discharge exposure may be due to the replacement of surrounding natural U columns with green metal.

Conversion Ratios - Pu

It was concluded from the data discussed in the preceding section that solid E-metal and lithium of the length ratio used would be near that needed for full-pile loading. The conversion ratios then will not need correcting for reactivity and can be based upon actual extraction numbers. The initial conversion of tritium is considered constant over the irradiation although some decay will occur.

Pu conversion ratios are rationalized with exposure by the formula

$$\text{Conversion in g/MWD} = C_0 e^{-bE}$$

where C_0 is the initial conversion in g/MWD and "b" is constant for a given pile, slug geometry and level of enrichment over an exposure range. E is the metal exposure and normally expressed in MWD/T. Accomplished production, then, can be translated into an initial conversion ratio by assuming calculated value for "b." This change from measured to initial values can also be calculated by measuring the amount of Pu and isotope levels. The initial conversions calculated by the two methods are given in Table No. 6.

TABLE NO. 6

INITIAL CONVERSIONS AND COLUMN EXPOSURES

	<u>Solid E-N Tube 2062</u>	<u>Solid E-N Tube 2162</u>	<u>Solid E-N Tube 2163</u>	<u>Cored E-N Tubes</u>
Exposure, MWD/T (from flow & outlet T)	682.3	703.3	731.5	833.0 Avg.
Exposure, MWD/T (Calc. by U-235, and Pu Differences)	689.3	699.8	741.3	776.2
Co g/MWD (using measured exposure and $g = ae^{-bE}$ where $b = 1.26 \times 10^{-4}$)	.834	.821	.783	.770
Co g/MWD (using Pu isotope analysis and fission to absorption cross-sections)	.844	.830	.788	.776

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The agreement between the two methods is reasonably good, but does show a consistent discrepancy in that all Co values from isotope analysis are consistently higher than those using accepted values for "b" values calculated using isotope measurements compared 1.42×10^{-4} vs. 1.26×10^{-4} accepted. Derivation of "b," and exposure isotope calculations used to derive the 1.42 are given in the Appendix.

Special Studies
Process and Reactor Development
IRRADIATION PROCESSING DEPARTMENT

LW Lang:bs

APPENDIX

I. Conversion Ratio

To verify the "b" value in the conversion ratio formula

$$g_{Pu}/MWD = ae^{-bE}$$

Pu absorption and fission is calculated to give a total Pu formed - See Appendix Table No. 1. The exposure is assumed that measured. The actual Pu divided by total Pu formed should be equal to e^{-bE} of the above equation. The fission to absorption cross-section for Pu is assumed 1.839.

If the higher value of "b" is accepted, the gain in initial conversions will be greater than indicated in Table No. 2, but the gain from reduced rate of Pu burn-out will be slightly less. Since the uncertainties between test calculated and theoretical calculated compensate near the exposure range of interest, 700-1000 MWD/T; the exact value of "b" is of little practical concern. However, the initial conversions are generally a convenient point of comparison. For this purpose, the lower initial conversions are listed.

Values used in previous documents are also given. The ratios determined by this load will not agree for the changed conditions of other loadings where additional refinements have been made. The Pu conversions for enriched I & E elements would be anticipated higher in roughly the same ratio than was obtained by I & E natural elements as compared to solid slugs. Also increased Li_6 content in the target element will reduce Al losses and increase conversion. Blanket loadings will likewise increase the pile conversion ratio. Modest extrapolations of the test results are made in Table No. 3 to give the possible order of magnitude numbers that might be expected under the different conditions.

The Pu conversions obtained in this type of loading would be expected lower than that obtained with solid E-metal columns used for enrichment. This might indicate present conversion ratios used for E-metal columns are conservatively low.

APPENDIX TABLE NO. 1

CALCULATION OF "b" VALUES USING ISOTOPE DATA

$$\text{Conv. Ratio} = ae^{-bE}$$

	Tube <u>2163</u>	Tube <u>2062</u>	Tube <u>2162</u>	<u>Cored</u>
Pu-239 (percent)	94.37	94.37	94.32	93.68
40-41-42 (percent)	5.64	5.64	5.70	6.33
Total Pu(g/T)	522	522	528	579
Pu-40-41-42	29.39	29.39	30.04	36.65
39 Fission	54.06	54.06	55.24	67.40
Total-Abs. & Fission	83.45	83.45	85.28	104.05
Total Pu	576.06	576.06	583.24	646.40
Exposure	731.5	682.3	703.3	832.98
gPu/MWD	.788	.844	.830	.776
Actual Total	.9055	.9063	.9016	.8956
Value eB	.0993	.0984	.1036	.1102
b = bE/E	1.36X10 ⁻⁴	1.43X10 ⁻⁴	1.48X10 ⁻⁴	1.32X10 ⁻⁴
Accepted Value (for truncated cosine)	1.26X10 ⁻⁴	1.26X10 ⁻⁴	1.26X10 ⁻⁴	1.24X10 ⁻⁴

II. Uranium Extraction and Analysis⁽⁶⁾

Analytical measurements have been completed and confirmed on the six special dissolvings of E-metal. The plutonium to uranium ratio, the concentrations of the plutonium isotopes, and the uranium-235 concentration were determined on each dissolving except one whose identity was lost in the dissolving process. The values and precisions are tabulated below. All precisions are at the 95% confidence level.

Tube 2163-B

Enrichment	.947%
U ²³⁵ after irradiation	0.8585%
g of Pu/ton of U	522 ± 7.5
Plutonium data	
Specific activity	1.598 x 10 ¹¹ d/m/gram
Isotopic content	
Pu ²³⁸	.01 % by wt.
Pu ²³⁹	94.37 %
Pu ²⁴⁰	5.24 %
Pu ²⁴¹	0.37 %
Pu ²⁴²	0.01 %

Tube 2062-B

Enrichment	.947%
U ²³⁵ after irradiation	.8652%
Grams of Pu/ton of U	522 ± 5.5
Plutonium data	
Specific activity	1.594 x 10 ¹¹ d/m/gram
Isotopic content	
Pu ²³⁸	.01%
Pu ²³⁹	94.37%
Pu ²⁴⁰	5.25%
Pu ²⁴¹	.35%
Pu ²⁴²	.02%

Tube 2162-B

Enrichment	.947%
U ²³⁵ after irradiation	.8640%
Grams of Pu/ton of U	528 ± 15
Plutonium data	
Specific activity	1.591 x 10 ¹¹ d/m/gram
Isotopic content	
Pu ²³⁸	.01%
Pu ²³⁹	94.32%
Pu ²⁴⁰	5.27%
Pu ²⁴¹	0.38%
Pu ²⁴²	0.02%

(6) HW-57277, Analysis of Special E-Metal Dissolvings, Harmon, K.M., August 26, 1958, Confidential-Undocumented.

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Mixture of tubes: 2068-B
2069-B
2168-B
2169-B

Enrichment	.947%
U ²³⁵ content after irradiation	.855%
Grams of Pu/ton of U	579 ± 6.2
Plutonium data	
Specific activity	1.625 x 10 ¹¹ d/m/gram
Isotopic content	
Pu ²³⁸	0.01%
Pu ²³⁹	93.68%
Pu ²⁴⁰	5.88%
Pu ²⁴¹	0.41%
Pu ²⁴²	0.02%

Tube 2063-B

Analysis of the dissolver solution of this tube and process tank volume measurements showed the dissolver to contain 250 pounds of uranium compared to 172 pounds of this tube charged. It must be concluded that the heel of Chalk River batch number 5 was not completely removed. The Chalk River material was processed in two dissolvings, the heel of the first not being removed. Since the solutions from these two cuts differed in uranium and plutonium properties, those in the final heel were highly questionable. So no attempt was made to calculate the properties of tube 2063-B.

Discussion of Analytical Methods

The uranium concentration of each solution was determined by making a TBP extraction with heavy salting and reading on the X-ray photometer (CPD method UX-4a). Since the acid-to-uranium ratio of these samples was unusually high, a synthetic sample similar to the E-metal solutions was used to test the method. The average recovery was 100.8% ± 1% on the synthetic. The U²³⁵ value was determined by the usual mass spectrometric method after purification by TBP extraction.

Concurrent with the uranium analyses, the plutonium alpha activity concentration of each solution was determined by the routine control laboratory method. (Total alpha activity is determined and corrections are applied for americium and uranium). To obtain the best precision and accuracy, 16 uranium and alpha determinations were made on each solution by 16 different individuals. The plutonium concentration was calculated from the specific activity of the plutonium and the alpha activity concentration of the solution.

Determination of Specific Activity

The specific activity of the plutonium in each batch was calculated from the isotopic concentrations and specific activities of the various isotopes. The concentrations of isotopes Pu²³⁹, Pu²⁴⁰, Pu²⁴¹, Pu²⁴² were determined with the thermal emission mass spectrometer while that of Pu²³⁸ was determined by

alpha energy analysis. Dowex 1 x 4, 100-200 mesh, anion exchange resin was used to separate and purify samples of plutonium for the isotopic analyses.

The calculations and constants used were as follows:

Specific activities used for isotopes

Pu ²³⁸	3.756×10^{13} d/m/g
Pu ²³⁹	1.363×10^{11} d/m/g
Pu ²⁴⁰	5.036×10^{11} d/m/g

Specific activity of sample

$$S. A. = \frac{(\% \text{ Pu}^{239}) \cdot 1.363 \times 10^{11} + (\% \text{ Pu}^{240}) \cdot 5.036 \times 10^{11}}{100 - (\% \text{ activity Pu}^{238})}$$

Pu²³⁸ percent by weight

$$\% \text{ Pu}^{238} = \frac{(S.A.) \cdot (\text{Pu}^{238} \% \text{ by activity}) \cdot 10^2}{3.756 \times 10^{13}}$$

Confirmation of Methods

The specific activity of plutonium as calculated from the fundamental isotopic data was found to compare very favorably with that determined by chemical analysis and alpha counting on the control laboratory instruments. Eight Redox L-6 samples on which the specific activity is routinely determined by chemical assay (PuX-3a and alpha counting) were analyzed for isotopic concentrations and the specific activities calculated from this data. The latter are compared to the laboratory specific activities below:

	<u>Ave. S. A.</u>	<u>Precision</u>
Chemical method	1.570×10^{11}	$\pm 1.5\%$
Isotopic method	1.571×10^{11}	$\pm 0.5\%$

In addition, the specific activities of the plutonium in the last two batches of E-metal tabulated were determined by a chemical assay and alpha count. On these solutions a coulometric titration (controlled potential coulometer) and alpha activity determination were made on aliquots of plutonium purified by anion exchange resin. The results are compared to the isotopic method below:

	<u>S. A. d/m/g</u>	
	<u>Coulometer count</u>	<u>Isotopic calculation</u>
Batch 5	1.620×10^{11}	1.625×10^{11}
Batch 6	1.855×10^{11}	1.840×10^{11}

Agreement between the two columns is within the precision of the coulometric titration (for a 200 g sample) which is estimated at $\pm 1\%$. The titration is considered a good confirmation of the isotopic method.

III. Lithium Extraction and Gas Analysis ⁽⁷⁾

A comparison of the theoretical He^3/He^4 ratio with that observed is given in Appendix Table II. The theoretical values of this ratio are computed for an assumed zero decay time of April 3, 1957 and which is the time at which the irradiation was four-fifths complete. The observed ratios of He^3 to He^4 for the eight extractions agree very well with the theoretical values, showing a maximum deviation of 2.5%.

The extraction conditions employed in this series were as follows: The slugs in batches of five, in their original cans were heated to 400°C and held for two to three hours at a pressure of less than 1 mm Hg, to remove absorbed gases and water from the outside surfaces of the cans. Neither tritium nor helium was found in this outgas fraction. The elements were then heated rapidly to a temperature of 610-620°C, where extraction was essentially completed below the melting point of the alloy. The temperature was then raised to 700°C and held for a minimum of 15 minutes, with a final furnace pressure of less than 100 microns. Any additional gas obtained upon fusion was added to the previous extraction gas, and the composite was mixed, measured volumetrically, and analyzed by mass spectrometer. Any tritium present in the furnace extraction gas as water vapor was frozen out in a cold trap refrigerated with liquid nitrogen, and subsequently thawed, diluted, and analyzed by decomposition with calcium and measured in an ion chamber.

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APPENDIX TABLE NO. 2
TRITIUM

<u>Tube</u> <u>No.</u>	<u>Slug</u> <u>Nos.</u>	<u>T₂</u>	<u>cc(STP)</u> <u>By Extraction</u> <u>He³</u>	<u>He⁴</u>	<u>cc(STP)</u> <u>Calc. (a)</u>
2062	2C- 3 -10 -17 -24 -31	537	50.5	1162	556
2168	4B- 5 -12 -20 -27 -34	586	62.1	1239	589
2068	4A- 3 -10 -17 -24 -31	578	66.6	1243	588
2169	3A- 3 -10 -17 -24 -31	563	66.7	1218	576
2063	6D- 5 -12 -20 -27 -34	559	74.5	1231	578
2163	1C- 3 -10 -17 -24 -31	551	77.4	1215	569
2069	1B- 5 -12 -20 -27 -34	570	83.9	1293	605

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APPENDIX TABLE NO. 2 (Cont'd)

Tube No.	Slug Nos.	cc(STP) By Extraction			cc(STP) Calc. (a)
		<u>T₂</u>	<u>He³</u>	<u>He⁴</u>	
2162	5D- 5 -12 -20 -27 -34	548	82.5	1206	562

(a) $\text{ccT}_2 \text{ present in slug at time of extraction} = \frac{\text{cc He}^4 - \text{cc He}^3}{2.00}$

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APPENDIX TABLE NO. 3

<u>Tube No.</u>	<u>Date of Extraction</u>	<u>He³/He⁴ x10²(a) (calc)</u>	<u>He³/He⁴ x10² (obs)</u>	<u>% Deviation</u>
2062	1-24-58	4.46	4.35	-2.5
2168	3-4-58	5.04	5.01	-0.6
2068	3-25-58	5.36	5.36	0.0
2169	4-9-58	5.58	5.48	-1.8
2063	5-6-58	5.98	6.05	+1.2
2163	5-28-58	6.28	6.37	+1.4
2069	6-12-58	6.50	6.49	-0.2
2162	6-26-58	6.72	6.84	+1.8

(a) Assuming zero time for decay was April 3, 1957, at which time irradiation was approximately 4/5 complete.