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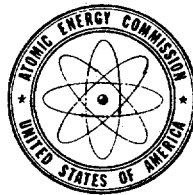
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**RICHLAND FIVE-YEAR
02 R&D PROGRAM**

PLUTONIUM-238



**RICHLAND OPERATIONS OFFICE
ATLANTIC RICHFIELD HANFORD COMPANY
BATTELLE-NORTHWEST
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June 30, 1968

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PRINCIPAL CONTRIBUTORS

D. W. Constable	DUN
G. F. Owsley	DUN
M. Szulinski	ARHCO

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PU-238 PROGRAMINTRODUCTION

There are three principal facets to the Pu-238 Program which are important to Richland. First, reactor neptunium production rates can be materially enhanced by judicious fuel management plans. Second, significant improvement in production efficiency and costs may be made if the Pu-238 production step (irradiation of Np-237) were sited at Richland. Further, Richland reactors have ample capacity, without reducing power, to irradiate all neptunium from government and commercial reactor sources which has been forecasted well into the 1980's. Third, a separate Pu-238 production process, that of irradiating Am-241 (obtained from power reactor plutonium returns), offers an attractive and competitive means to materially supplement Pu-238 from the Np-237 route. The first two of these aspects of the Pu-238 Program are being pursued actively, particularly in light of firm and predicted requirements for Pu-238. Technological development for irradiating and processing Am-241 will be performed in the near future.

To enhance neptunium production, it is important that uranium rich in U-236 be put back into the reactors on an expedited basis. It is also important that the materials with the highest U-236 concentrations be placed in the N Reactor because of its more advantageous effective U-236 cross section. However, substantial gains can also be realized by increasing the U-236 concentrations at the other three reactors. With the present economic basis, enhancing U-236 concentration in natural and slightly enriched fuels by re-enriching with higher enriched uranium and recycling increases the ultimate cost of the Pu-238. Consequently, the degree to which the U-236 inventories can be maintained in the reactor system requires detailed consideration of the incremental effect on the feed cycle cost. However, a further source of U-236 is available from slightly enriched uranium irradiated in other reactors, such as that available from NFS. Thus, substantial increases in neptunium production within economic limits can be realized through the prudent management of U-236 supplies which are becoming available.

An efficient process for the irradiation of neptunium to produce Pu-238 has been worked out and demonstrated. It has been determined that maximum production levels are attained when the exposure time is adjusted to yield a plutonium product containing approximately 90 percent Pu-238. The relatively short (50-60 days) out-of-reactor time, demonstrated at Richland, is an equally important factor in obtaining maximum Pu-238 production levels. As a result of achieving the programmatic goals to date, the technology is now established for full-scale production of Pu-238 using Richland neptunium with an optimum relationship between the fabrication, irradiation, and reprocessing steps.

The technical development of producing Pu-238 from the irradiation of Am-241 has been re-scheduled to start in FY-1969. The immediate need for this capability is small as the result of the rather minor quantities of Am-241 which will be available in the near future. However, this production route is particularly attractive because the Pu-238 should contain negligible Pu-236 contaminant and can potentially be processed in a manner to yield very pure Pu-238. Initiation of a test irradiation program is planned for FY-1969.

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SCOPE AND OBJECTIVES

The objective of this program is to increase the national production of Pu-238 in as efficient and economical a manner as practical, while giving full attention to satisfying the short-term market requirements for Pu-238. This program is directed to the utilization of the Richland production complex in meeting this objective.

To maximize the production of neptunium, principal effort is placed on better quantification of the conversion rates of Np-237 from U-236, fuel management techniques for maximizing the U-236 in the fuel streams within economic limitations, and optimum utilization of uranium fuel in the four reactors consistent with production requirements.

In producing Pu-238 from the irradiation of Np-237, several factors must be considered in accelerating the conversion rate while maintaining efficiency. Emphasis has been placed on optimizing the neutron flux level, the related target and reactor load design, and the exposure per cycle. Further reprocessing steps have been implemented which achieve a minimum out-of-reactor time between successive recycles of the Np-237, and reduce the neptunium and plutonium losses. Production studies and proposals have also considered utilizing existing facilities for separation and fabrication in order to minimize costs associated with a production scale program. Those factors involved in optimizing the process have been established through the completed development and demonstration programs. Principal future work will be directed to improving the various neptunium and Pu-238 production steps, and reducing the Pu-236 contaminant level.

The significant availability of Am-241 forecasted for the 1970's from power reactor plutonium tails suggests the irradiation of Am-241 as an attractive supplemental route for producing Pu-238. Further, the Pu-238 produced from Am-241 should be free of the Pu-236 contaminant. The Cm-242 obtained in this process may also have significant value as an energy source. Am-241 target fabrication, irradiation, and separation techniques need to be identified and developed. The development is related to the work performed on the Transplutonium Program (Mission 3) and the Np-237 processing studies. Integrated facilities for separations and fabrication may be feasible. In fact, it may be possible to use common separation and fabrication facilities for the production of Cm-244, Pu-238 from Np-237, and Pu-238 from Am-241.

INCENTIVES

The operation of the Richland production reactors results in the production of substantial quantities of Np-237 as a by-product. Since the production of Pu-238 is limited by the national availability of Np-237, there is considerable incentive for adapting feasible and economically attractive methods for enhancing the production of Np-237.

The short-term incentive for moving part of the neptunium irradiation to Richland stems primarily from the increased production capabilities. Richland's long-term

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incentive for participating in the conversion of Np-237 to Pu-238 stems primarily from characteristics of the Richland complex which probably make Richland the optimum site for the ultimate production of Pu-238. Advantages at Richland include a favorable thermal neutron energy spectrum (which results in a significantly lower ratio of Pu-238 destruction neutron cross section to Pu-238 formation neutron cross section compared to thermal reactors with lower moderator temperature); the availability of existing facilities for processing the neptunium and Pu-238 with only very small capital modifications for low throughput and only moderate capital modifications for high throughput capability; the capability for a closely integrated fabrication, irradiation, and separation process with relatively low operating cost (which permits minimum out-of-reactor time for recycled neptunium); and the reactor capability to vary the flux level to optimum values. A further important consideration is that due to the type of charge-discharge scheduling and the potential for charging and discharging neptunium elements during operation, the flexibility of the Richland reactors permits nearly continuous flow of the neptunium at a precise exposure.

In addition, there is an advantage to the AEC for capability to process neptunium at a second site. This will provide the flexibility to mesh Pu-238 production with other programs in an optimum manner as the Pu-238 program increases in the future. Also, second site participation can be expected to result in efficiency and cost gains as a result of the competition between the two sites.

Production of Pu-238 from Am-241 provides a potential for substantially increased Pu-238 production capability which will be significant in the middle and late 1970's when quantities of Am-241 become significant from spent power reactor fuels. Being free of the Pu-236 contaminant, Pu-238 produced by this route may be particularly attractive for medical purposes. It is expedient that the technology be developed now to provide well-integrated expansion into this production area.

PROGRESS DURING REPORT PERIOD

A test scale irradiation of 72 Metal with 400 ppm U-236 was performed in B Reactor to obtain a more exact measure of the effective U-236 cross section. Analysis is in progress. From previous measurements it was not possible to differentiate with sufficient accuracy between the neptunium produced from the U-238 ($n, 2n$) source and the U-236 (n, γ) source. This delineation is important for accurate assessment of Np-237 production rates with recycled uranium containing higher concentrations of the U-236. A similar test with 95 Metal containing 1000 ppm U-236 was charged into a K reactor.

Efforts on U-236 management in N Reactor are continuing. Currently, 0.947% U-235 fuel containing 350 ppm U-236 is being routinely charged into the reactor. A production test has been initiated, and a portion of the fuel charged, to obtain neptunium production data for fuel containing 1145 ppm U-236 at planned exposures up to 4000 Mwd/ton. With the assistance of the Hanford U-236 management

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committee, all suitable feed materials high in U-236 received at the feed site are being directed into the N Reactor fuel stream.

Demonstration of an improved irradiation technique using kilogram quantities of neptunium was completed in the Hanford reactors. A 50 percent higher flux level was achieved through special loading techniques in a K reactor and charge and discharge of a portion of the neptunium elements was accomplished while the reactor was operating. The K reactor test yielded a wide range of exposures in the irradiation which produced a Pu-238 quality range of 80-95 percent. Also, a portion of the neptunium was irradiated in specially prepared elements containing graphite rather than aluminum as the diluent for the purpose of isolating the effect of aluminum on the production of the Pu-236 contaminant.

Evaluation of the neptunium irradiation in N Reactor was completed and the final report of the production test was issued. Included in the report was an analysis of the exposure (74 to 147 full power days) and neptunium concentration (45 and 70 g nep/foot) on total production. The total neptunium burnup ranged from 5% to 24.7%, giving a product purity range of 85.1% to 95.2%. The data and analysis from these pilot scale programs provide the basis for proposing a continuing Pu-238 production program at Hanford.

Measured production parameters for both reactor types are presented in Figures I and II and are compared to predicted values. Analysis of the Pu-236 production parameters is in progress. However, initial data indicates that Pu-236 concentrations as low as 0.4 ppm were obtained in the graphite matrix elements.

Fabrication processes were evaluated and developed to provide for the production of neptunium-graphite target elements. Fabrication and irradiation variables of neptunium-impregnated graphite targets and neptunium oxide-graphite compacted pellet targets were studied. The neptunium oxide-powder graphite pellet process was selected as an acceptable process for these materials. Redesign of target end contours was completed to minimize misalignment of the neptunium target elements during irradiation. Pressure tests of the redesigned target cans were completed to confirm target strength data.

Studies were performed of alternate materials for use as a matrix for neptunium in Pu-238 production schemes. Materials studied include MgO, PbO, ZrO, ZnO, and metallic magnesium. The objective of the studies is to provide an alternate for powdered graphite which is unreactive to the reactor coolant and compatible with target fabrication, irradiation, and chemical separations requirements.

Neptunium irradiated in the N and KE Reactor demonstration tests was separated by the ion exchange process in Pacific Northwest Laboratory hot cell facilities and processed to oxide in ARHCO laboratory facilities. The product was of high quality, and initial shipments to Mound Laboratory for further evaluation have been made. Typical product analyses are given in Tables I and Ia. Considerable "know how" was developed in the course of the program. An adaptor for the Pu-238 nitrate transfer can was developed and used such that no contamination incidents resulted during transfer of solutions between areas, and operations

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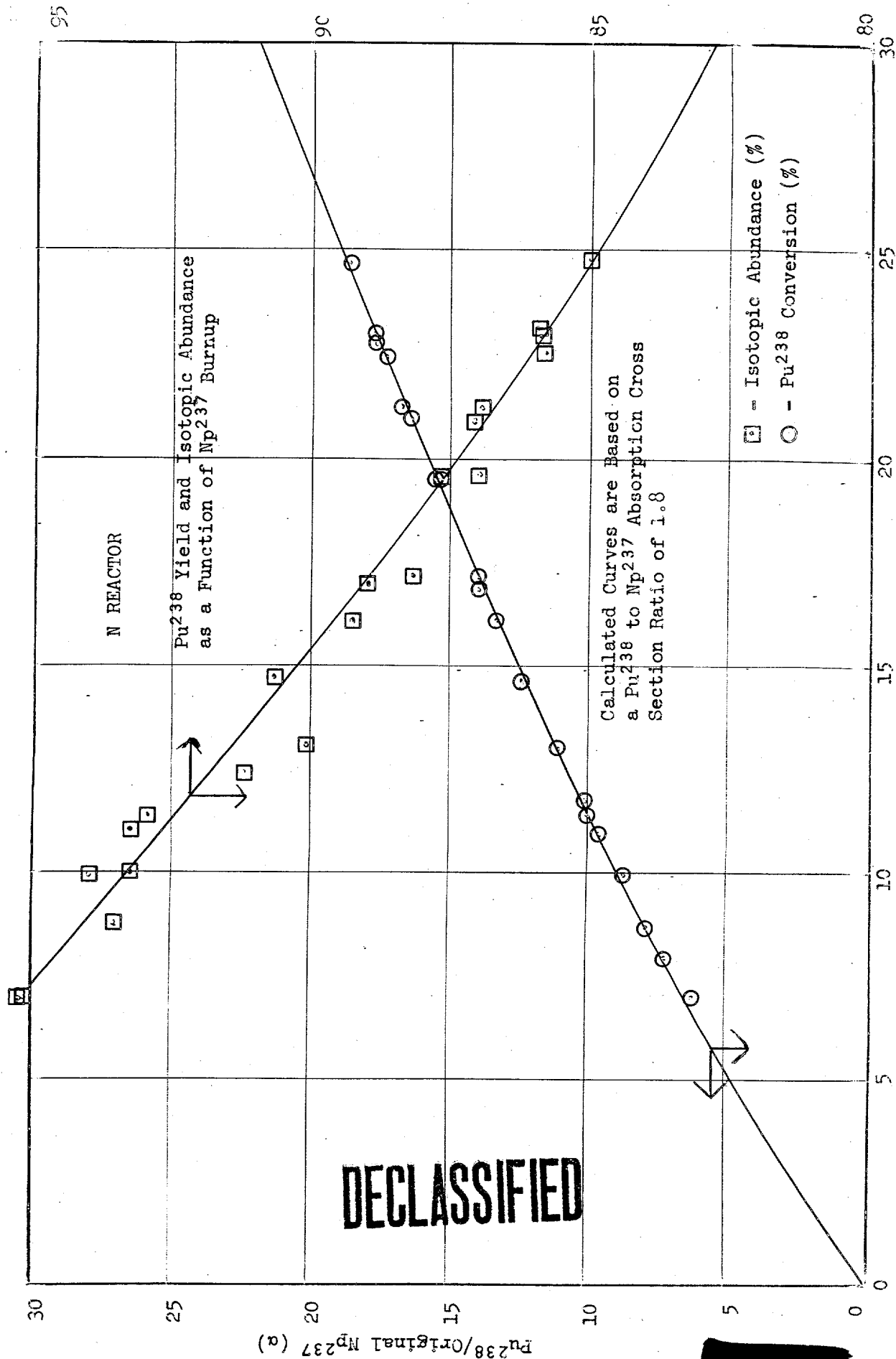


FIGURE 1
Np²³⁷ Destroyed/Original Np²³⁷ % (1-8)

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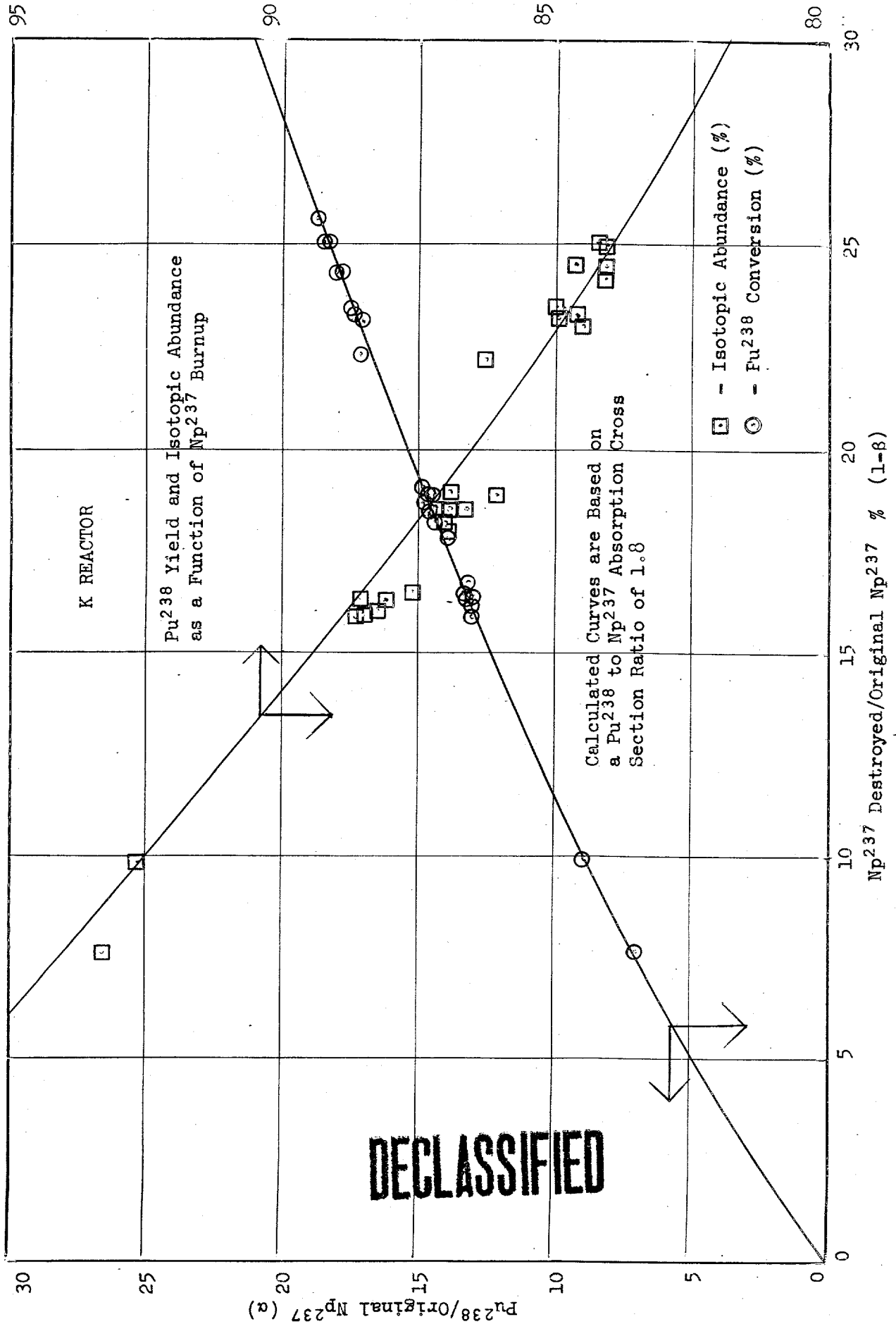


FIGURE 2

TABLE I

PU-238 PRODUCT QUALITY

Shipment Batch	1	2	3	4	5
Grams, Oxide	91.075	109.10	112.995	106.76	58.03
Assay, % Pu	87.98	85.78	87.62	85.46	87.28
Bulk Density	1.62	2.33	1.19	1.36	1.42
Neutron Activity, n/sec/g 238($\times 10^4$)	2.80	2.57	2.58	2.61	2.57
<u>Impurities, p/m 238</u>					
Neptunium	477	2400	99	120	1200
Thorium	3290	<535	4800	3629	2070
Uranium	153	5	280	1000	65
<u>Fission Products, $\mu\text{Ci/g 238}$</u>					
Zr-Nb 95	1.24	11.6	18.6	35.3	120
Ru-Rh 106	1.72	<0.4	<2.37	<17.2	<100
<u>Loss on Ignition, %</u>					
To 450°C	-	0.44	0.38	0.71	0.31
450 to 700°C	-	0.35	0.54	0.12	0.08
<u>Isotopic Content</u>					
Pu-236 (ppm)	0.5	0.5	0.6	0.6	0.5
-238, wt%	92.57	87.18	86.13	86.5	86.5
-239, wt%	6.82	10.38	11.11	10.95	10.85
-240, wt%	0.68	1.91	2.17	1.98	2.03
-241, wt%	0.11	0.48	0.51	0.49	0.54
-242, wt%	0.01	0.061	0.08	0.07	0.07

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TABLE Ia

PU-238 PRODUCT QUALITY

Metallic Impurities - ppm
(Emission Spec)

	<u>Shipment</u>				
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
Ag	<1	100	20	5	200
Al	<5	5	200	100	0
As	<20	<20	<20	<20	<20
B	20	-	5	5	2
Be	<1	1	<1	<1	-
Bi	<1	-	<1	<1	<1
Ca	5	20	20	10	-
Cd	<1	<2	5	5	5
Cr	<10	5	<10	<10	<10
Cu	<1	<2	1	2	2
Fe	<25	33	350	53	465
Ge	<5	<10	<5	<5	<5
K	<5	10	<5	10	20
Li	<5	<10	<5	<5	<5
Mg	<1	50	2	2	1
Mn	<1	<5	<1	<1	<1
Mo	<5	<5	<5	<5	<5
Na	2	200	5	1	20
Ni	<5	50	<5	<5	<5
P	<100	<100	<100	<100	<100
Pb	5	-	100	200	20
Si	340	10	480	220	550
Sn	<2	-	10	2	2
Ti	<5	-	10	10	-
Tl	<1	<1	<1	<1	<1
V	<100	<10	<100	<100	<100
Zn	<5	<10	100	5	100

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for precipitation and conversion to oxide proceeded with minimal difficulty or radiation exposure. The radiation from neptunium and Pu-238 was further characterized. Wastes from aged Pu-238 were processed and 370 mgs of the first daughter, U-234, were recovered with an isotopic purity of 88 percent.

The chemical processing considerations of neptunium oxide, irradiated in a graphite matrix, were studied. Potential processes for grind-leaching, electrolytic disintegration-leaching, chemical oxidation and burning were considered, and burning was selected for processing the material irradiated in the K Reactor test. Evolution of radioiodine as an organic iodide was identified as a potential problem with short-cooled graphite targets.

The ion exchange process was further refined to result in a high quality product; however, it was indicated that a two-cycle process will be required. Solvent extraction work was initiated, and equipment fabricated and installed to allow high-radioactivity level development and demonstration of flowsheets in support of production facilities. Low-level testing indicated di butyl butyl phosphonate (DBBP) to be a potentially desirable solvent as well as tributyl phosphate (TBP) which is most useful in existing production plants.

Production modes of processing large quantities of irradiated neptunium in both the Redox and the Purex Building were studied. Tentatively it appears that processing through the Purex process on a campaign basis is feasible and, likely, the economic optimum method of producing large quantities of Pu-238 at Hanford. Production of the plutonium and neptunium oxides and fabrication of target elements by compaction of neptunium oxide and aluminum powder in the Z Plant facilities appears to be feasible and consistent with cycle requirements.

Detailed plans were initiated for fabricating a few elements of americium in an aluminum diluent for irradiation. The objective of this small program is to establish the burnup and buildup parameters in a Hanford reactor leading to Pu-238 formation.

EVALUATION OF EFFORT

Reliable Np-237 production predictions with recycle uranium have been established, thus providing a firm basis for Np-237 production calculations and economic evaluations of uranium management modes designed to increase Np-237 output.

Pu-238 production rates from irradiation of neptunium in the Hanford production reactors have been established by experimental measurements obtained from pilot scale irradiations. An improved method for irradiating neptunium utilizing a higher neutron flux level and a facility for the charge and discharge of the target elements during operation was successfully demonstrated. These accomplishments, coupled with the Np-237 - Pu-238 conversion demonstration, have provided the technology for moderate scale Pu-238 production program proposal using existing Hanford facilities. The demonstration program also provided a base to extrapolate a larger production program in the mid and late 70's.

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Studies of alternate matrix materials for neptunium targets were initiated and proceeded at a relatively low level of effort. This work has progressed satisfactorily in light of the interest in and need for such alternates. Optimization of the graphite process and evaluation of other materials can be readily accelerated as a changing demand might dictate.

Chemical processing "know how" for separations and production of neptunium and Pu-238 oxides has been developed and a significant quantity of Pu-238 oxide has been produced with no major difficulty. The radiation and shielding requirements for these materials have been characterized. Processes for separating Pu-238 from irradiated NpO_2 -graphite targets have been developed and processing of a test batch has been initiated. A significant quantity of U-234, the daughter of Pu-238, has been isolated.

Solvent extraction processes for large-scale separation of Pu-238 are under development, and equipment for high radioactivity level demonstration work has been installed and is ready for operation. Production studies, completed and in progress, indicate that, with appropriate modifications, either a Redox or Purex Plant could be used for large scale production. It is likely that campaign operation throughout, assuming that adequate process time is available and appropriate equipment modifications have been made, will result in lower incremental cost. Such campaign operation, however, will not maximize Pu-238 production rates such as could be realized by rapid turn-around in continuous processing.

The R & D effort to firm up the technical basis of the large scale production will require further work to optimize the target design with respect to separations considerations, as well as minimum Pu-236 production; and to demonstrate the solvent extraction separations flow sheet.

BUDGET PERIOD PLANS

The K reactor irradiation and analysis of the 95 Metal containing 1000 ppm will be completed. The results will extend the U-236 level in recycle uranium for which Np-237 production rates can be calculated reliably. The effective U-236 cross sections will be evaluated from the completed irradiation of 400 ppm U-236 in 0.72 percent enriched uranium. The completion of the irradiation of high U-236 Mark 1-C fuel (see the Basic Production Program Document) will provide data to better quantify the effective U-236 cross section in the N Reactor. It is planned that an irradiation of neptunium using either a reduced quantity of aluminum diluent or an alternate diluent will be initiated as a continuation of the program to investigate methods for reducing both the Pu-236 contaminant level in Pu-238 and the separations costs of the neptunium elements. The irradiation will also be designed to further quantify the effectiveness of various techniques for reducing the Pu-236 contamination such as minimizing the source of fast neutrons from adjacent fuel tubes which trigger the $\text{Np-237 (n,2n) Np-236}$ reaction and for reducing Np-238 fissions by lowering the flux level.

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Evaluation of alternate matrix materials will continue with some consideration being given to the reduction in bulk amounts of matrix material to be used in the targets. Toward this end, hollow pellets of target material and other target geometries will be considered. A small scale test irradiation of a new target concept will be prepared for irradiation as process development or product demand might require.

Ion exchange separations studies to improve process performance with respect to losses, decontamination resin stability, and economics will be continued. Solvent extraction work in support of a large-scale separation will be continued with particular emphasis on demonstration of a flowsheet for the Purex Plant. Development of in-line instrumentation for process control will be initiated. Processing studies on alternate target materials and designs will be conducted, and the economic incentives for graphite as a target matrix will be explored further. Engineering development in support of a plant design will be conducted. Processes for processing americium for Pu-238 recovery via Cm-242 decay will be tested.

Continuous calcination of neptunium nitrate to neptunium oxide will be tested. Processes for purification of Pu-238 oxide from daughters of Pu-236 will be scouted and developed.

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MILESTONE SCHEDULE

Complete characterization of Pu-236 contaminant levels in Hanford-produced Pu-238	July, 1968
Complete design and initiate test fabrication of improved neptunium target elements to minimize Pu-236 production	September, 1968
Complete irradiation and analysis of improved neptunium target elements	March, 1969
Identify, for project action, optimized Pu-238 production mode at Hanford and complete supporting documentation	July, 1969
Complete evaluation of irradiation tests for establishing effective U-236 cross sections in Hanford reactors	June, 1969
Initiate Am-241 target element fabrication and irradiation	July, 1968
Complete evaluation of Am-241 irradiation	June, 1969

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STATISTICAL SUMMARY SCHEDULE

	<u>Fiscal Year</u>				
	<u>1969</u>	<u>1970</u>	<u>1971</u>	<u>1972</u>	<u>1973</u>
<u>Dollars (in thousands)</u>					
DUN Reactors	75	50			
ARHCO	<u>275</u>	<u>250</u>			
Total	350	300	310	320	330
<u>Man Years</u>					
DUN Reactors	2.2	1.3			
ARHCO	<u>4.6</u>	<u>4.7</u>			
Total	6.8	6.0			
<u>Equipment (in thousands)</u>					
DUN Reactors	0	0			
ARHCO	<u>30</u>	<u>75</u>			
Total	30	75			

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FY 68	FY 69	FY 70	FY 71	FY 72	FY 73
	TEST CONT. CALCIN. OF NP	DEMO CONT. CALCIN. OF NP			
CHARACTERIZE PU-236 LEVELS	ION EXCH. STUDIES				CONTINUOUS EFFORT TOWARD IMPROVED NP-PU SEPARATIONS
DES. & INIT. TEST FAB TRGTS. TO MIN. PU-236	IRRAD & ANALY. OF IMPROVED TRGT.				
	IDENTIFY OPTIMIZED PU-238 PROD. MODE				CONTINUOUS DEVELOPMENT TO MINIMIZE PRODUCT RADIATION LEVEL
					CONTINUOUS EFFORT TOWARD IMPROVED PU-238 PRODUCTION
	EVALUATE TEST FOR U-236 CROSS SECTIONS				CONTINUOUS EFFORT TOWARD INCREASING NEPTUNIUM PRODUCTION
	Am-241 TRGT. FAB IRRAD & EVAL.				CONTINUOUS EVALUATION OF Am-241 CONVERSION TO PU-238

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