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RICHLAND FIVE-YEAR 02 R&D PROGRAM

PLUTONIUM-238

RICHLAND OPERATIONS OFFICE
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RICHLAND FIVE-YEAR

O2 R&D PROGRAM

PU-238 PROGRAM

RICHLAND OPERATIONS OFFICE
DOUGLAS UNITED NUCLEAR, INC.
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PU-238 PROGRAM

PRINCIPAL CONTRIBUTORS

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

[redacted]

RICHLAND FIVE-YEAR

O2 R&D PROGRAM

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PU-238 PROGRAM

INTRODUCTION

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 can 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 carried out 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 concentration be placed in the N Reactor because of its more advantageous U-236 cross section. However, substantial gains can also be realized by increasing the U-236 concentrations at the other four 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 and continued study of the conversion of Np-237 to Pu-238 to attain an optimum relationship between the fabrication, irradiation, and reprocessing steps.

The technical development of producing Pu-238 from the irradiation of Am-241 is scheduled to start in FY-1968. 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. Initiation of an irradiation program of test quantities is planned for FY-1968.

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 five reactors consistent with production requirements.

To satisfy the short term Pu-238 market, it is extremely important that the irradiation of Np-237 be initiated early. The multi-kilogram test recently completed provides adequate technology to base a decision for starting the production program.

In producing Pu-238 from the irradiation of Np-237, several factors must be considered in accelerating the conversion rate while maintaining efficiency. Emphasis will be placed on optimizing the neutron flux level, the related target and reactor load design, and the exposure per cycle. Further reprocessing steps will be implemented which achieve a minimum out-of-reactor time between successive recycles of the Np-237, and reduce the neptunium and plutonium losses. Optimizing the process will also consider utilizing existing facilities for separation and fabrication in order to minimize costs associated with a production scale program.

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. In addition, 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; the same chemical separations are involved and the proposed methods of fabrication and irradiation are nearly identical to those for Np-237. 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 potential. Richland's long term incentive for participating in the conversion of Np-237 to Pu-238 stems primarily from the probability that characteristics of the Richland complex may make Richland the optimum site for the ultimate production of Pu-238. Advantages at Richland include the availability of existing facilities for processing the neptunium and Pu-238 with only very small capital modifications for low through-put and only moderate capital modifications for high through-put capability; the capability for a closely integrated fabrication, irradiation, and separation process with relatively low operating cost, and 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. However, it is expedient that the technology be developed now to provide well-integrated expansion into this production area.

PROGRESS DURING REPORT PERIOD

A. NP-237 PRODUCTION

A continuous effort has been underway: to obtain experimental data on neptunium formation in the reactors; to devise and test theoretical models for predicting neptunium conversion ratios; and to utilize basic production numbers in fuel cycle management computational models for enhancing Np-237 formation.

The experimental effort has centered on planning and charging some carefully controlled tests to determine neptunium production yields from the various uranium isotopes as a function of initial U-236 assay and exposure. Several special irradiations currently in the reactors such as the 210 metal-lithium test at N and KW Reactors and high exposure fuel from one of the low pressure reactors will supplement the information to be obtained from the special tests.

Recent correlations between computed and experimental data have been confined to the computer codes MOFDA and POSEIDON. Both codes include U-236 self shielding and can be used to predict neptunium production to within experimental errors for

standard and overbore fuel geometries for the low pressure reactors and N Reactor fuels. Some recent data have been evaluated and are presented in Table I.

To achieve still better agreement between measured and computed U-236 buildup and neptunium irradiation at higher exposures, the mathematical models are being updated.

A set of equations based on computed results was developed for all current fuel element models. These relationships will permit evaluation of neptunium formation from the various uranium isotopes as a function of initial U-236 concentration and exposure. Any available experimental data can be incorporated to readjust the constants in these equations and improve their reliability. This set of relationships will be used extensively in generating accountability tables for neptunium production.

The basic production numbers calculated by the theoretical models were used in numerous fuel management studies enhancing neptunium production and U-236 buildup in the K and small reactors. A special emphasis was placed on investigating the increases in neptunium production due to natural uranium recycle with associated economic considerations.

B. PU-238 PRODUCTION

To provide the basis for extrapolating full scale production capability, the AEC in FY-1966 established a program to irradiate 4.4 kg of neptunium at the Richland site. These tests have been completed and a full-scale production proposal[1] has been made. In addition, a new program has been initiated involving the irradiation of approximately 3 kg of neptunium in the KE Reactor.

Irradiation

Results of the reactor irradiations are summarized in Tables IIA and IIB for N Reactor and Table III for K Reactor. The proposal[1] to produce Pu-238 by irradiating Hanford generated neptunium is based on burnup and conversion ratio data verified by the irradiation testing program. Since no enhancement scheme has been assumed, this Pu-238 production level becomes a reliable quantity.

To further study the Np-237 - Pu-238 production mode, a new program involving the irradiation of 3 kg of neptunium in the KE Reactor is in progress. A zone was established in the reactor which provides for a 50% higher flux level in the neptunium to demonstrate an accelerated production rate. In addition, a large fraction of the neptunium has been charged during operation using equipment developed in the early 1960's.

Since the Pu-236 content in Pu-238 creates a radiological problem, methods are being investigated for decreasing the Pu-236 concentration. Analytical studies indicate that the high energy gamma rays from the n,γ reaction with aluminum lead to a γ,n reaction with the Np-237 which makes a significant contribution to

[1] DUN-AOP-44

the production of Pu-236. To isolate, and thus measure, this contribution elements with graphite as a diluent have been prepared for irradiation.

Fabrication

The K Reactor irradiations utilized targets made from sintered aluminum metal with neptunium oxide, while N Reactor irradiations used an aluminum-neptunium alloy. The favorable experience with the alloy and the off-gassing problems with the sintered elements have led to the adoption of the alloy for future irradiations using the aluminum diluent. Problems associated with the radioactivity from neptunium have been well characterized with the processing of the short out-of-reactor time neptunium from the N Reactor irradiations. The activity levels from the target elements are displayed graphically in Figure 1. It has been concluded that it is feasible to process large quantities of neptunium as long as recycled neptunium is not handled outside shielded facilities prior to 45-50 days after it leaves the reactor.

A few neptunium elements containing graphite as a diluent were fabricated. One method impregnated neptunium nitrate into a pre-formed graphite rod which was subsequently heated to convert the nitrate to oxide. The other technique was to sinter neptunium oxide with graphite. These elements will be irradiated early in FY-1968.

Reprocessing

The major developmental effort has taken place in the separations area. Principal problems involve developing flow sheets and techniques which would minimize the losses of the plutonium and neptunium, prevent iodine release to the atmosphere, provide for a satisfactorily high decontamination factor for fission products, and provide a through-put capacity using existing facilities with minimum capital cost. Substantial progress has been made in developing the flow sheets and evaluating the capability for handling the different parts of the program in existing facilities.

A chemical flowsheet was issued for the processing of irradiated Np-Al targets in the Purex processing plant. This scheme of operation would be useful for processing irradiated neptunium in relatively large lots since the entire plant is applied to the task and extensive flushing would be required to effect a turnaround. Although technology is available for processing neptunium and plutonium in the standard Purex process, considerable extrapolation from existing data is required in the formulation of a neptunium-plutonium separation and purification flowsheet. Solvent extraction studies are expected to be initiated by fiscal year end.

The irradiated Np-Al targets from N Reactor were processed by Battelle Northwest in C-Cell of the 325-A Building. Neptunium-aluminum target dissolution data were obtained, and flowsheets for separations and purification of Np-237 and Pu-238 from these target materials by ion exchange was demonstrated with targets cooled for as short a time as 15 days. Control of radioiodine evolution during the mercury-catalyzed nitric acid dissolution proved adequate, and the HgI_2 precipitation

[redacted]

process for removal of radioiodine before ion exchange processing was demonstrated satisfactorily in the course of the demonstration test. The results agreed well with those predicted from earlier laboratory work.

The short-term cooled material was processed to oxide by the Plutonium Chemistry Laboratory as part of the test irradiation currently in progress at the KE Reactor. Radiation from the Np-238 had decreased sufficiently at the forty-first day after reactor shutdown to allow oxide production via direct calcination. Radiation fields were somewhat high for routine production; however, good shielding data were obtained. During these demonstrations, a batch direct calcination concept for the neptunium nitrate was tested successfully and work toward defining the process chemistry of Pu-238 removal from neptunium nitrate was accomplished. The measured radioactivity with time of a target element fabricated from this material is given in Figure 1.

Laboratory studies were made of iodine behavior during and following caustic-nitrate dissolution of Np-Al targets. It was found that little radioiodine was evolved during element dissolution (< 2%), and that containment during alkaline feed stream manipulation presented little problem. Most of the radioiodine (> 90%) was converted to the elemental form immediately upon neutralization and acidification of the feed, thereby presented a major containment problem. One contact of the acid feed with 30% TBP-NPH transferred 99% of the radioiodine to the organic layer. Radioiodine removal from the acid feed by solvent extraction with benzene or forced volatilization was 97% and 85% effective, respectively.

The concept that iodine can be controlled by melting the target under conditions that evolve iodine and trapping it by a suitable absorbent is under test. In preparation for laboratory studies of fluoride volatility processes, experimental equipment has been purchased and assembled for studying fluid bed hydrocholorination reactions of Np-Al target materials (as well as U-Zr fuel materials under Mission 6). Preliminary experiments are underway. Building facilities have been designed for use of fluorine. As a corollary to work performed under Mission 1 concerning the recovery of americium from aged plutonium metal by fractional distillation, calculations were made to evaluate the feasibility of separating Pu-238 from neptunium metal by a similar process. A single simple distillation does not appear attractive; however, a fractional distillation of plutonium from neptunium appears feasible.

In support of a proposed, interim semi-production campaign, studies were done which showed the feasibility of using idle facilities, with minor modifications, in Z Plant to convert neptunium nitrate to oxide and to fabricate Np-Al target elements clad in aluminum. Studies are being conducted to determine the feasibility of converting the Pu-238 nitrate to oxide in similar facilities at Z Plant for this semi-production case. With existing capabilities it is necessary to rely on PNL facilities for chemical processing of the irradiated targets.

EVALUATION OF EFFORT

During FY-1967, processes for fabrication, irradiation, and separation of Np-237 have been identified and tested. Also, conversion rates of neptunium to Pu-238 have been established experimentally. With these accomplishments, the objectives for the year have been met. The technology has been developed to the point that a production proposal for the processing of Np-237 at Richland has been prepared and submitted.

The Am-241 program has been delayed due to the press of other higher priority work. This planned delay is considered acceptable due to the limited availability of Am-241 until the mid-1970's.

BUDGET PERIOD PLANS

Irradiation and Fabrication

The neptunium production rates from high exposure 72 metal (up to approximately 1800 Mwd per ton) and high U-236 concentrations (400 and 1000 ppm) will be determined from irradiations in progress and planned. These will provide more extensive and rigorous knowledge of the effective incremental U-236 cross sections and resonance self-shielding in the Richland reactors. The abundance of reliable experimental data which will become available will permit extensive correlation and reevaluation of the validity of the computational models such as POSEIDON and MOFDA for high exposures and large fuel elements. Also, the neptunium production in 210 metal will be measured.

With the possible use of fully enriched fuel in the DUN reactors, considerations will be given to experimental and computational work associated with neptunium formation in such fuel elements.

The Np-237 irradiation program has been extended to better define the conversion parameters, utilize a higher flux irradiation, demonstrate the application of the operational charging and discharging of neptunium targets, and in the separations area demonstrate the rapid reprocessing of neptunium and recovery of Pu-238 on a more nearly production-scale basis.

The conversion rates over a wider range of exposure will be identified. Irradiations to date were limited to burnups of neptunium of 25 percent or less. These will be extended to the 30-35 percent burnup range. Also, the effect on the Pu-236 contamination level of replacing the aluminum diluent will be measured. Plans are being made to investigate even higher flux levels than those in the current test in the KE Reactor. Irradiation with higher enriched uranium fuel will be investigated both analytically and experimentally. The processes will be investigated using alternate diluents. These include magnesium oxide and zirconium in addition to graphite. To develop the Pu-238 program completely and to resolve the optimum production scheme, an R & D program will be established for producing other types

of target elements. Included in this effort will be the development of a neptunium oxide element. These studies will attempt to show the fabrication rate, neptunium lost during fabrication, efficiency of chemical separation, and the cost associated with the various types of elements. Test scale irradiations of Am-241 will be performed to identify key technological problems and feasible processes, and to measure conversion rates.

Reprocessing

Solvent extraction studies in support of the Redox flowsheet will be completed. Further studies of anion exchange purifications will be made to optimize the flowsheet for CPD purposes. Engineering studies will be made to find the optimum way to process irradiated neptunium targets at Richland. Processing studies will be conducted on alternate target materials; e.g., mixed ceramics, Np-graphite, or possibly magnesium and zirconium alloys. These will have the goals of simplifying chemical processing or of resulting in Pu-238 of higher quality; that is, with lower Pu-236 content. Support will be continued for processing irradiated neptunium targets from the reactor demonstration programs.

Demonstration runs will be made verifying the flowsheets for Np-Al alloy scrap recovery. These runs will include alloy dissolution and neptunium purification by anion exchange. Special emphasis will be placed on recovery techniques for the cryolite process skull material. Processes for continuous direct calcination of neptunium nitrate will be scouted. Technology will be developed for the preparation of Am-241 target element alloy for use in the Cm-242 - Pu-238 production test core and for milking Pu-238 from Cm-242 nitrate solution.

TABLE I

Np-237 Production Data

Experiment	Exposure Mwd/t	Experimental U-236 Buildup ppm	Experimental Np-237 Prod g/ton of U	POSEIDON Calc. U-236 Buildup ppm	POSEIDON Calc. Np-237 Prod g/ton of U	MOFDA Calc. U-236 Buildup ppm	MOFDA Calc. Np-237 Prod g/ton of U
Recycle E metal 03E	785	159	3.77 ± .23	171	3.93	166	3.98
	722	153	3.46 ± .23	158	3.55	147	3.59
	721	142	3.54 ± .40	156	3.55	147	3.59
	658	136	3.11 ± .22	141	3.28	150	3.31
1/2" overbore	395	70	1.77	87	1.77	79	1.82
C6N	752	130	3.69	160	3.84	141	3.74
N Reactor 0.946 Mark I	2138	360	15.45	425	15.70	374	13.77

[redacted]

[redacted]

TABLE IIA

EXPOSURE DEPENDENT PRODUCTION PARAMETERS
MEASURED ALONG COLUMN N-0856 (70g-Np/ft)

Equivalent Full-Power Days*	(1-β),%	α,%	ε,%	Purity,%
	8.0	7.3	91.7	95.2
	10.0	8.9	88.7	93.4
90.0	11.5	10.2	88.3	93.0
	11.0	9.8	89.0	93.4
74.5	10.0	8.9	89.5	93.9
	8.8	8.0	90.2	93.7
44.0	7.0	6.3	90.7	95.4

TABLE IIB

EXPOSURE DEPENDENT PRODUCTION PARAMETERS
MEASURED ALONG COLUMNS N-0858 and N-0860 (45g-Np/ft)

Equivalent Full-Power Days*	(1-β),%	α,%	ε,%	Purity,%
174.8	24.7	18.9	76.5	85.1
	23.3	18.0	77.1	86.0
162.4	23.0	17.9	78.1	85.9
	22.6	17.5	77.3	85.8
	21.4	17.0	79.4	87.0
135.0	20.8	16.6	79.8	87.2
	19.7	15.6	79.6	87.0
	19.6	15.8	80.9	87.8
	17.3	14.2	81.8	88.3
104.0	17.1	14.2	82.8	89.0
	16.2	13.6	83.9	89.4
76.6	14.7	12.6	85.8	90.8
	13.1	11.2	85.7	90.2
67.7	11.8	10.2	87.2	91.4

*The equivalent full-power days were only calculated on the elements containing flux monitor wires. They were obtained by flux weighting the actual column residence time.

TABLE III

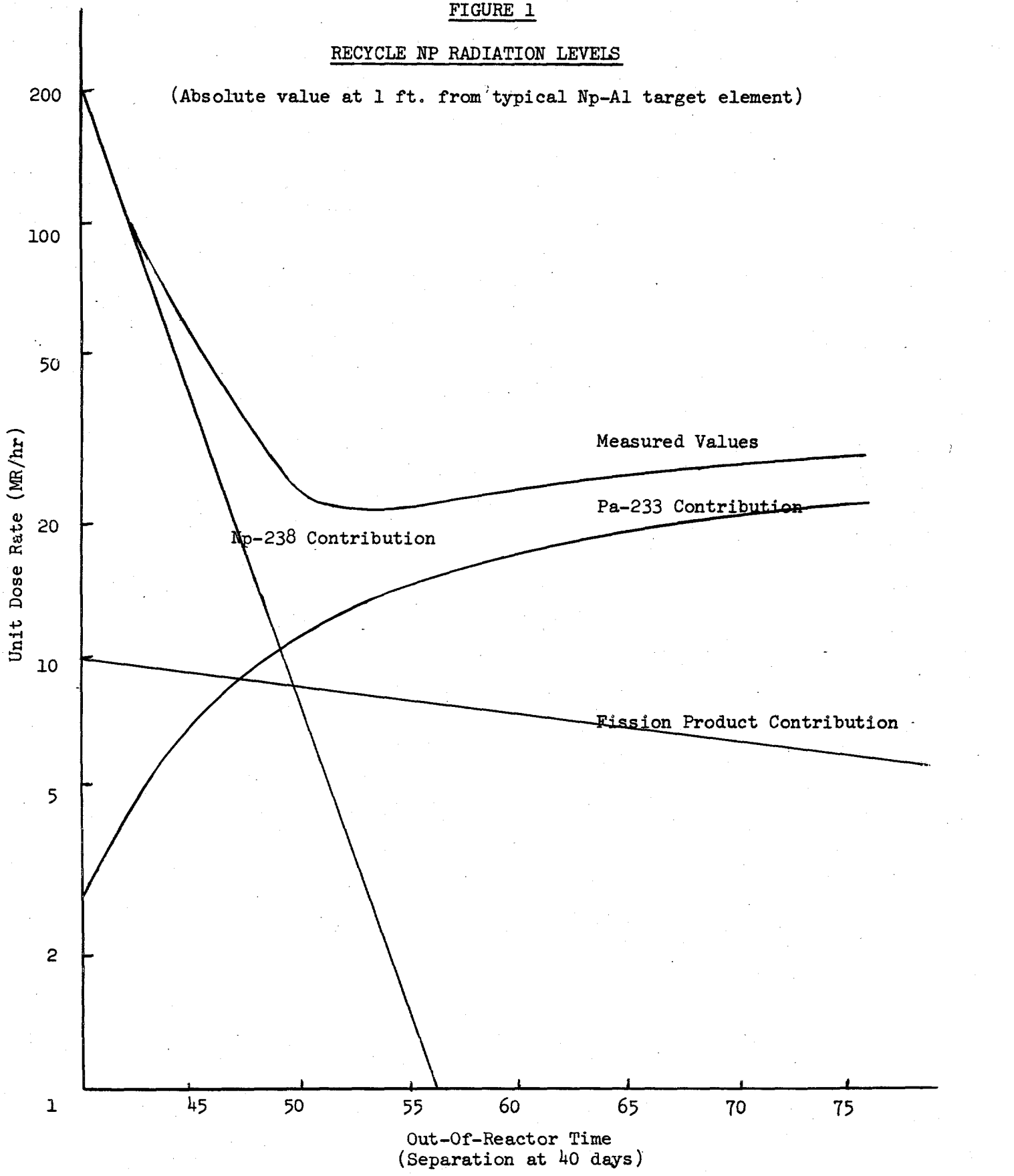
NEPTUNIUM IRRADIATION PARAMETERS IN KW REACTOR

Np Conc. Gms/ft	Full Power Days	(1-β),%	α,%	Purity,%
38	101	14.9	12.5	89.4
38	101	15.2	12.8	89.3
38	189	25.7	18.1	82.1
38	189	24.8	17.6	82.3
153	189	17.3	13.7	86.8
153	189	16.8	13.9	88.2

FIGURE 1

RECYCLE NP RADIATION LEVELS

(Absolute value at 1 ft. from typical Np-Al target element)



MILESTONE SCHEDULE

Initiate Development of Ceramic Neptunium Target	January, 1968
Complete 3 kg K Reactor Neptunium Irradiation and Analysis - Including Identification of Pu-236 Formation Parameters	March, 1968
Complete Evaluation of 400 ppm U-236 Program to Establish Effective U-236 Cross Sections	April, 1968
Initiate Irradiation of Am-241	April, 1968
Establish and Demonstrate Separation Flow-Sheets for Advanced Target Forms	April, 1969
Establish Optimized Target Designs	June, 1969
Establish Technical Criteria for Neptunium Target Separation Facility	June, 1969
Establish Technical Criteria for Neptunium Target Fabrication Facility	June, 1969

STATISTICAL SUMMARY SCHEDULE

	Fiscal Year				
	1968	1969	1970	1971	1972
Dollars (in thousands)					
B, C, and K Reactors	120	80	70	70	60
N Reactor	100	100	200	200	200
ARHCO	180	240	400	300	200
Total	400	420	670	570	460
Man Years					
B, C, and K Reactors	0.8	1.6			
N Reactor	1.6	1.6			
ARHCO	3.9	5.5			
Total	6.3	8.7			
Equipment					
B, C, and K Reactors	5	0			
N Reactor	0	0			
ARHCO	17	30			
	22	30			

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