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AEC RESEARCH AND DEVELOPMENT REPORT

ORNL METAL RECOVERY PLANT

PROCESSING CLEMENTINE REACTOR

FUEL ELEMENTS: TERMINAL REPORT

J. L. Matherne

OAK RIDGE NATIONAL LABORATORY
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ORNL METAL RECOVERY PLANT PROCESSING CLEMENTINE REACTOR FUEL ELEMENTS: TERMINAL REPORT

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CONTENTS

0.0 ABSTRACT

This report presents data obtained from processing 33 Clementine Reactor fuel elements in the ORNL Metal Recovery Plant to recover approximately 15 kg of plutonium and 0.16 g of americium.

1.0 SUMMARY

The Los Alamos Clementine Reactor fuel elements were processed in the Metal Recovery Plant to recover a total of 14,622 g of plutonium and 0.16 g of americium. A total of 14,716 g of plutonium was shown by analysis to be contained in the ORNL dissolver solutions; Los Alamos S.F. data had indicated the amount to be 15,005.5 g.

The plutonium was recovered by dissolving the fuel elements, separating the plutonium from americium by a modified Purex solvent extraction cycle, and isolating the plutonium on a resin bed. Americium was recovered by neutralizing the aqueous waste stream from the plutonium extraction column, separating the americium from ionic contaminants by solvent extraction and isolating the americium on a resin bed.

The plutonium product from solvent extraction was concentrated tenfold by sorption on Dowex 50 resin followed by batch elution with 6.0 M nitric acid. The product solution from the resin column, containing an average of 51 g of plutonium per liter, was shipped to the Rocky Flats site for further processing.

The gross beta and gamma fission product decontamination factors from plutonium were 250 and 175, respectively. The relatively low decontamination factors were due to a low concentration of fission products in feed solution. All plutonium recovered from the Clementine fuel met fission product specifications for recovered plutonium.

A total of 0.15 g of americium was recovered.

The total cost of recovering Clementine Reactor fuel was $56,834, or $3.88 per gram of plutonium recovered.

2.0 INTRODUCTION

The Los Alamos Fast Breeder (Clementine) Reactor used plutonium fuel elements. After it had operated from late 1947 to early 1953, one of the fuel elements ruptured, contaminating the coolant and other fuel rods, and the reactor was shut down. The Atomic Energy Commission requested that the ORNL Metal Recovery Plant recover the plutonium and associated americium from the 33 elements. These elements were nickel-coated, gallium-alloyed plutonium rods encased in mild steel cans. The rods had been fabricated from 200 Mwd/ton and 400 Mwd/ton plutonium and had been irradiated 4 Mwd in the Clementine reactor.

This material was processed in the ORNL Metal Recovery Plant from September 15 to October 15, 1954. The objective of this program was to recover the plutonium and americium sufficiently pure and concentrated for further processing at the Rocky Flats site and the University of California Radiation Laboratory, respectively.

3.0 DESCRIPTION OF THE PROCESS

3.1 History and Description of Clementine Fuel Elements

The Clementine fuel elements consisted of gallium-alloyed plutonium slugs; each slug was approximately 0.64 in. in diameter and 5.5 in. long, weighed approximately 455 g, and was coated with a 0.003-in. layer of nickel. The nickel-coated plutonium rod, along with a 0.345-in.-thick 0.647-in.-dia (about 30 g) normal uranium wafer, was enclosed in a mild steel can. No bonding material was used in the fabrication of these pieces. Information received from Los Alamos[1] indicated that 6 kg of the fuel elements had been fabricated from 200 Mwd/ton plutonium while the remaining 9 kg had been made from 400 Mwd/ton material.

The rods were irradiated for a total of 4 Mwd in the Clementine reactor, which was operated at an average power level of 20 kw, from late 1947 until January, 1953. Based on the radiation history of the plutonium, it was estimated that approximately 3 g of americium was associated with the 9 kg of 400 Mwd/ton plutonium.

3.2 Chemical and Equipment Flowsheets

The chemical flowsheet[2][3][4] (Figs. 3-1 and 3-2) was designed to recover the plutonium and americium from the Clementine fuel elements at a purity and concentration acceptable to Rocky Flats and Livermore, respectively. The major items of process equipment and the flow of process solutions are shown schematically in Figs. 3-3 and 3-4.

Processing consisted in dissolution of the fuel elements; one solvent extraction cycle for plutonium separation, decontamination, and isolation; and neutralization of the aqueous waste stream from the plutonium extraction cycle followed by a solvent extraction cycle for americium separation and isolation.

A fuel element was removed from the shielded shipping container to a small lead pig and placed in the dissolver dry box. The slug was then transferred to a small stainless steel basket, which was secured to the dissolver hatch with a stainless steel chain, and lowered into the dissolver. Fifty gallons of 13 M nitric acid and sufficient concentrated hydrofluoric acid to obtain 0.05 M HF were added to the dissolver, and the solution was refluxed at maximum temperature (approximately 125°C) for 12 hours. After refluxing, the dissolver solution was allowed to cool, was steam-jetted to one of two feed tanks (A-6 or A-8), and sufficient 2 M aluminum nitrate was added to the solution to give an extraction feed solution which was 1.3 M in aluminum nitrate and 4.0 M in nitric acid. Aluminum nitrate was required to promote the extraction of americium.

The adjusted feed solution was pumped to the plutonium extraction column (IA) in which the feed solution flowed countercurrently to a stream of solvent (tributyl phosphate in Amsco diluent). The plutonium was extracted by the solvent. The plutonium-bearing solvent, after being scrubbed with dilute nitric acid, cascaded into the stripping column (IB) in which the plutonium was stripped by an aqueous solution of hydroxylamine sulfate. The plutonium-bearing aqueous stream (IBP) from the IB column cascaded through the IBP surge pot and sand filter and then through one of three resin columns, operated in parallel, where the plutonium was sorbed on Dowex 50 resin. When one resin column was loaded with approximately 500 g of plutonium, another column was put on stream, and the plutonium was eluted from the loaded unit with a mixture of nitric acid and sulfamic acid.

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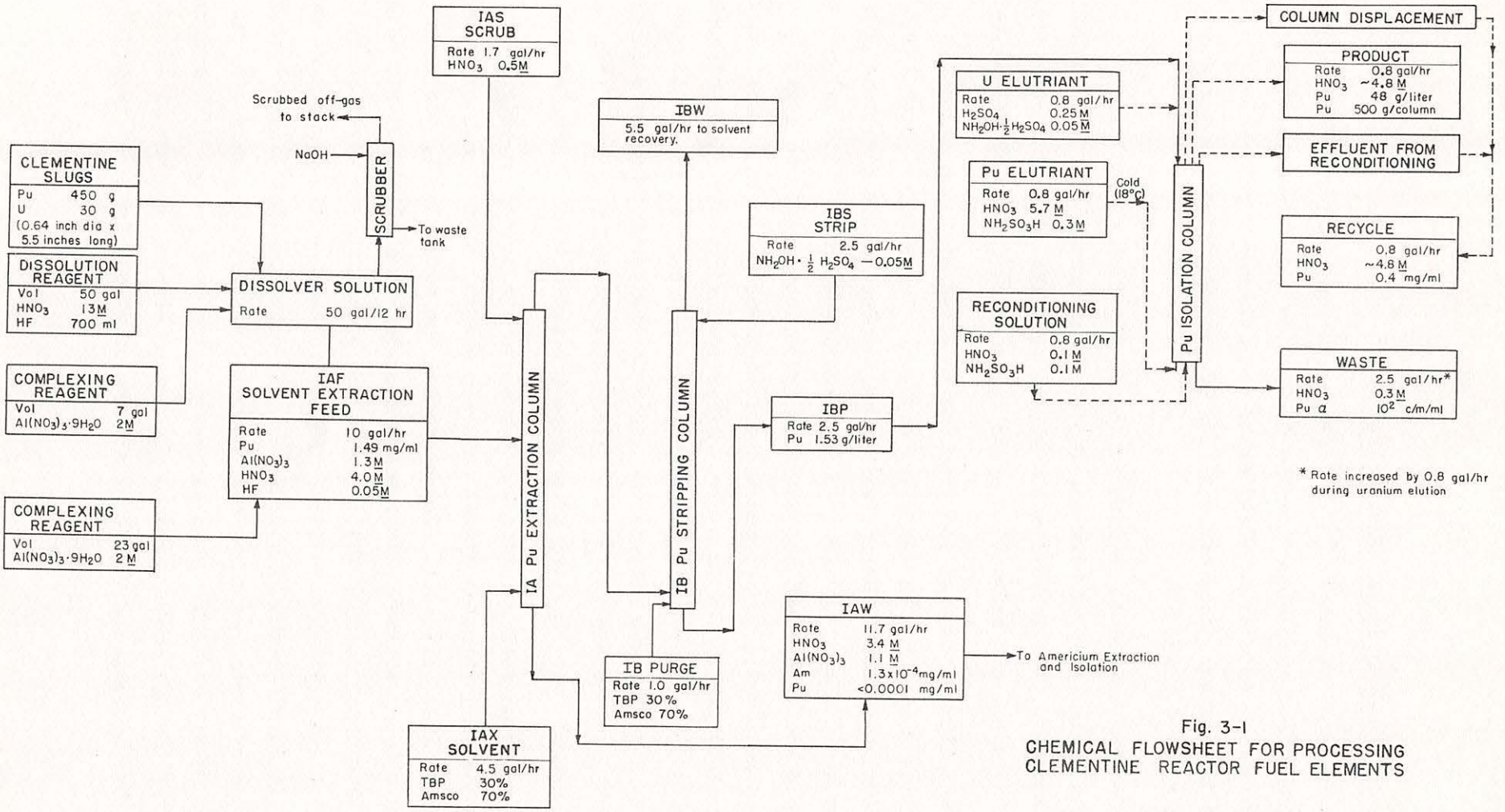

* Rate increased by 0.8 gal/hr during uranium elution

Fig. 3-1
CHEMICAL FLOWSHEET FOR PROCESSING
CLEMENTINE REACTOR FUEL ELEMENTS

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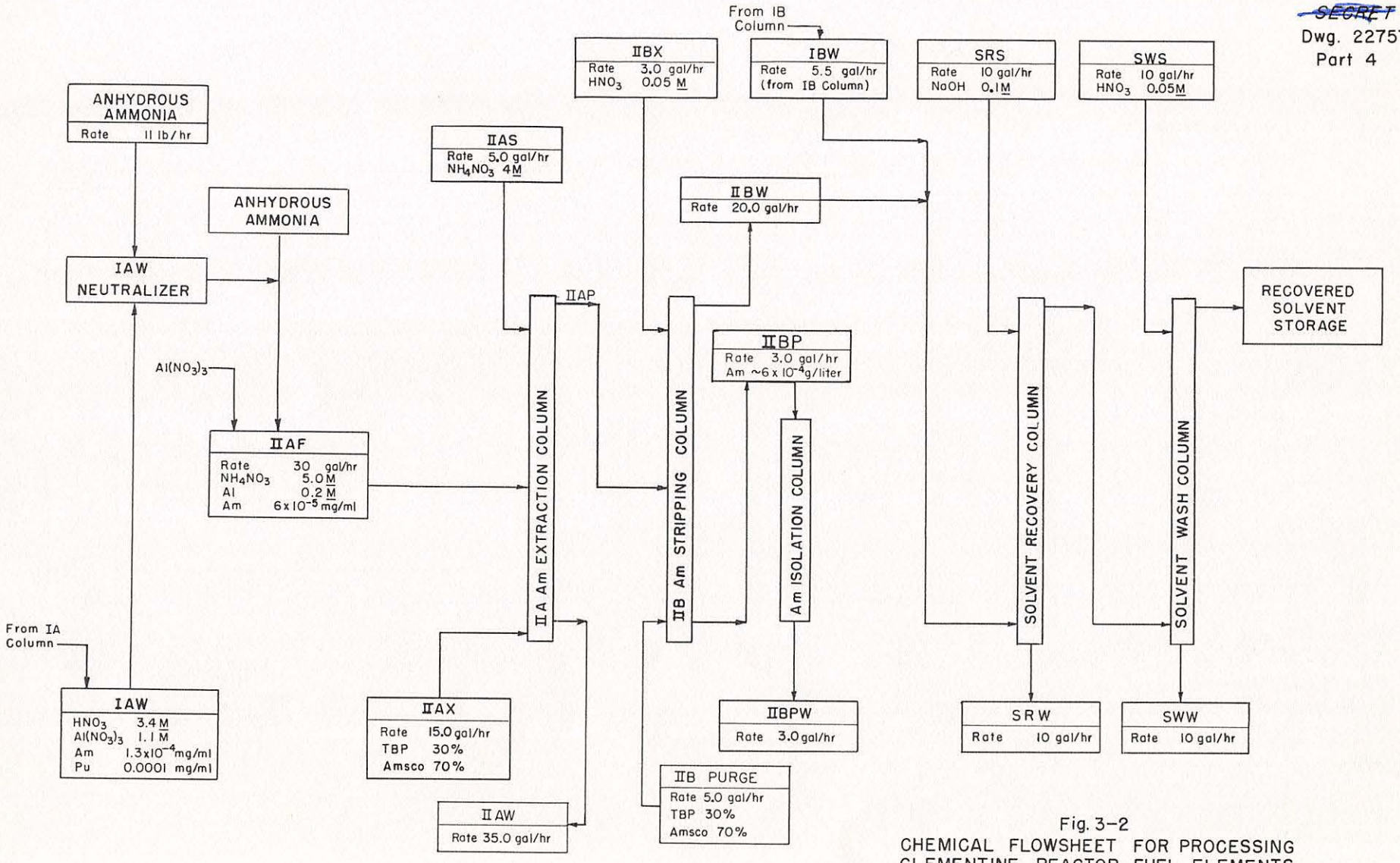

Fig. 3-2
CHEMICAL FLOWSHEET FOR PROCESSING
CLEMENTINE REACTOR FUEL ELEMENTS

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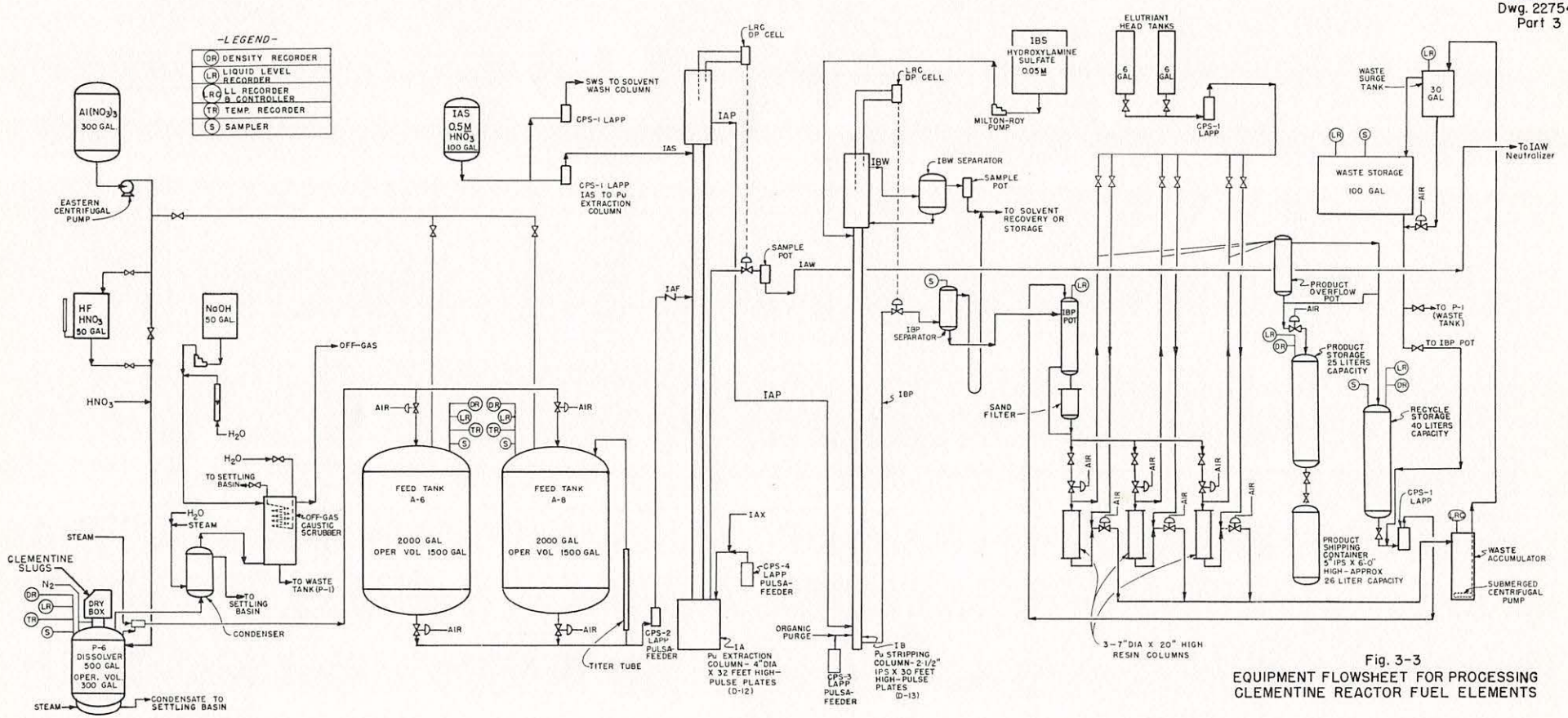

Fig. 3-3
EQUIPMENT FLOWSHEET FOR PROCESSING
CLEMENTINE REACTOR FUEL ELEMENTS

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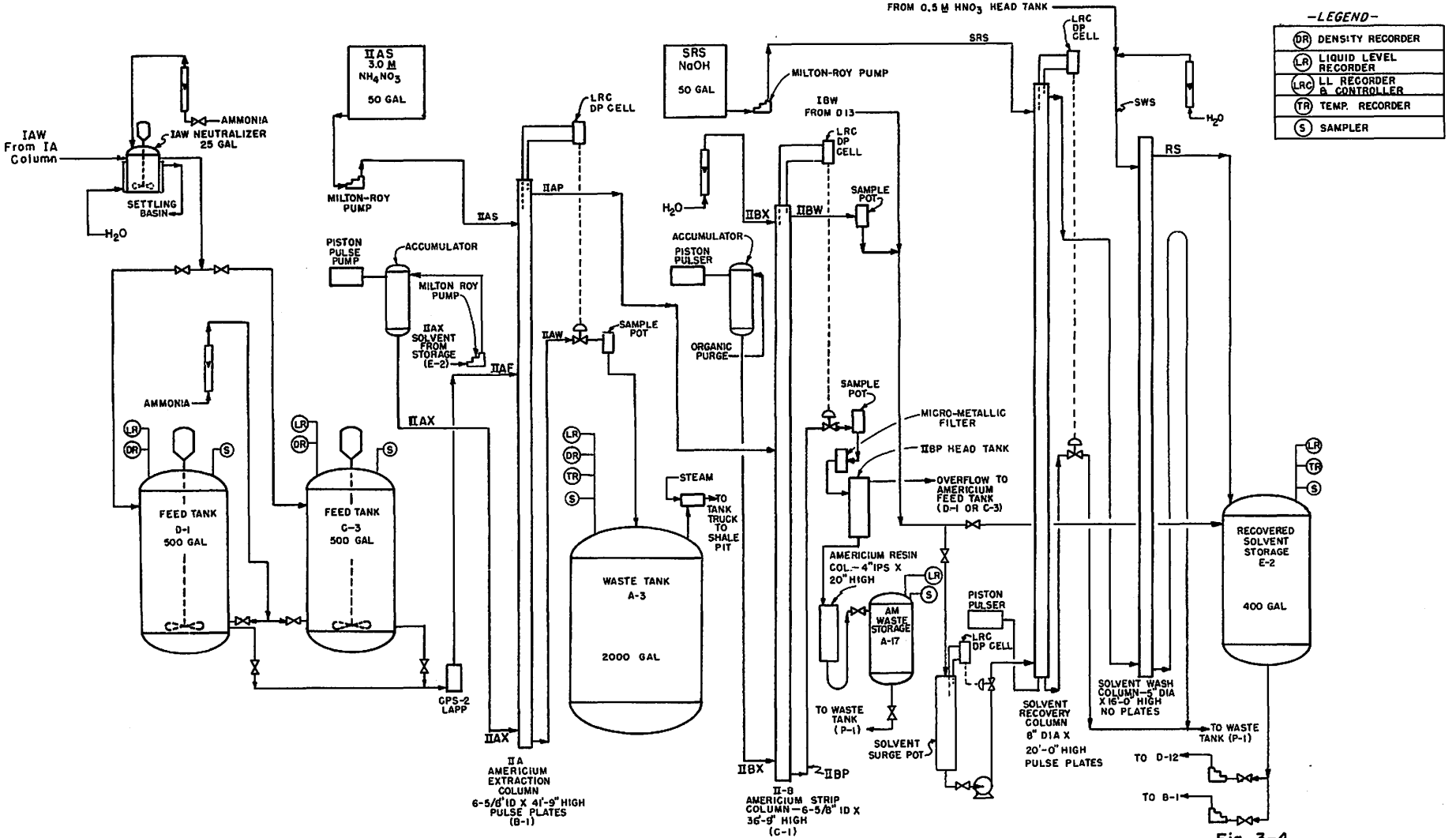

Fig. 3-4
EQUIPMENT FLOWSHEET FOR PROCESSING CLEMENTINE REACTOR FUEL ELEMENTS

Prior to elution of the plutonium, the uranium that was retained on the resin was removed from the column with sulfuric acid solution. The $Pu(NO_3)_3$ product solution was transferred to shipping containers and shipped to the Rocky Flats site.

The americium was recovered from the aqueous stream from the plutonium extraction column (IA). This was neutralized (pH 2) with anhydrous ammonia, 90 per cent of the neutralization being accomplished continuously in the neutralizer and the remainder in one of two feed tanks (C-3 or D-1). The neutralized solution was pumped to the americium extraction column (IIA) in which the americium was extracted by a stream of solvent (TBP-Amsco). The americium-bearing solvent, after being scrubbed with ammonium nitrate solution, overflowed into the IIB stripping column. The americium was stripped from the solvent with dilute nitric acid. The acidic americium solution (IIBP) flowed to the IIBP surge tank, from which it was pumped through a Micro Metallic filter to a resin column for sorption of the americium on Dowex 50 resin. The americium was eluted from the loaded resin column with ammonium acetate -- acetic acid solution.

The solvent was continuously contacted with sodium hydroxide solution and then washed with acid prior to being reused.

The principal process waste, aqueous waste from the americium extraction column was collected in a storage tank (A-3) and disposed of in the ORNL shale pit. Other process wastes, principally from the resin columns and solvent treatment, were combined in a tank (P-1) and jetted to the tank farm (tank W-5 or W-6).

4.0 PLUTONIUM CYCLE

4.1 Plutonium Recovery and Material Balance

Analyses of dissolver solutions indicated that the 33 Clementine reactor fuel elements contained a total of 14,716.06 g of plutonium, or 98 per cent of the 15,005.52 g shown on the S.F. transfer document from Los Alamos. A total of 14,622.54 g of plutonium was recovered as product, representing a recovery of 99.4 per cent, based on dissolver analysis.

4.2 Dissolution

Results of the first slug dissolving (solution analysis and visual inspection at 4-hr intervals) indicated that the slug would completely dissolve in 12 hr (Fig. 4-1). The majority of subsequent dissolutions were complete in 12 hr, although, 16 to 20 hr of boiling was required for complete dissolution in a few instances.

The detailed dissolving data are presented in Table 8-1.

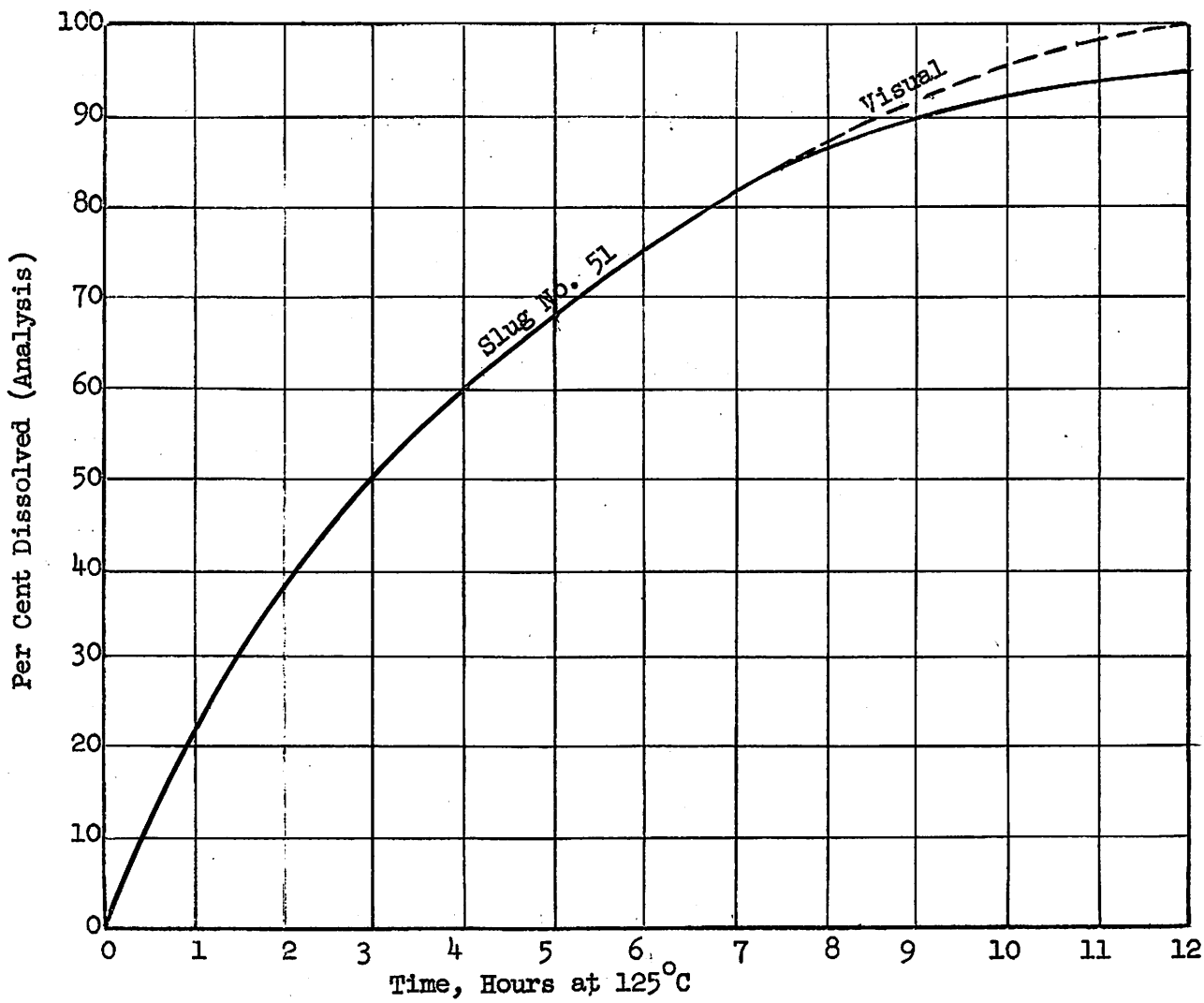

Fig. 4-1

Amount of Clementine Slug Dissolved in 13 M Nitric Acid (125°C) as a Function of Time

4.3 Feed Adjustment

Laboratory studies indicated that the concentration of ammonium nitrate in the feed solution to the americium extraction column would be insufficient to salt the americium into the solvent. Therefore, sufficient 2.0 M aluminum nitrate solution was added to the dissolver to make the feed solution 1.3 M in aluminum nitrate and approximately 4.0 M in nitric acid. (The salt solution was added to the dissolver because it was easier to add it to this tank than to the americium cycle feed tank).

4.4 Extraction Cycle

The Metal Recovery Plant extraction columns operated at an on-stream efficiency of 88 per cent. An average of 578 g of plutonium was processed per operating day.

4.5 Flowing Stream and Composite Losses of Plutonium

The plutonium loss from the first cycle was 0.14 per cent, based on analyses of flowing stream samples taken at 12-hr intervals. The plutonium loss for the whole program was 91.4 g, or 0.63 per cent, based on samples taken from waste tanks (P-1 and A-3) in which all process waste streams were collected. Individual stream losses are presented in Table 4-1.

Table 4-1

First Cycle Plutonium Losses

Stream	Loss, % of IAF Pu
IAW	0.003
IBW	0.135
IBPW	0.001
Total	0.139
Composite	0.625

4.6 Plutonium Fission Product Decontamination

Radiochemical analyses of feed and product solutions indicate that the plutonium gross beta and gamma decontamination factors were 250 and 175, respectively. These relatively low factors were due to low fission product activity in the feed solution. All plutonium recovered met fission product specifications for highly purified plutonium.

The individual fission product decontamination factors are shown in Table 4-2.

Table 4-2

Plutonium Fission Product Decontamination Factors

Constituent	Feed Analyses, cts/min/ml	Feed Analyses, cts/min/mg Pu	Product Analyses, cts/min/ml	Product Analyses, cts/min/mg Pu	Decontamination Factor
Gross β	9.65×10^5	1.22×10^6	2.40×10^5	4.89×10^3	250
Ru β	4.44×10^5	5.57×10^5	5.79×10^4	1.18×10^3	470
Zr β	3.2×10^3	4.06×10^3	226	5	800
Nb β	1.58×10^3	2.0×10^3	484	10	200
Ce β	2.6×10^5	3.3×10^5	4.24×10^4	861	400
TRE β	3.7×10^5	4.69×10^5	5.06×10^4	1.03×10^3	450
Zr β	3.2×10^3	4.06×10^3	---	---	---
Gross γ	404 mv/ml	511 mv/ml Pu	145 mv/ml	295 mv/ml Pu	175

4.7 Plutonium Product Analyses

The plutonium product from elution of the resin columns had an average plutonium concentration of 50.84 g per liter, an average nitric acid concentration of 4.23 M, and an average uranium content of 0.73 per cent of the plutonium content. The specific activity of the plutonium product averaged 7.49×10^7 alpha counts/min per milligram of plutonium, calculated from the ratio of the gross alpha concentration (counts/min/ml) to the plutonium concentration (mg/ml) as determined by the potentiometric method of analysis. Analyses of the individual product solutions are presented in Table 8-2, Appendix.

At the request of the AEC, the plutonium product from the 200 Mwd/ton material was kept separate from the 400 Mwd/ton material for separate processing by Rocky Flats. The contents of nine containers were analyzed for plutonium isotopic content (see Table 4-3).

Table 4-3

Isotopic Analysis of the Plutonium Contained in Nine Shipping Containers

Container No. PP-	Plutonium Isotopic Content, wt % 239	240	241	Approximate Irradiation Level, Mwd/ton
101	98.09 ± 0.05	1.84 ± 0.04	0.07 ± 0.02	Mixed (200 + 600)[a]
102	98.48 ± 0.05	1.48 ± 0.05	0.04 ± 0.01	200
103	98.59 ± 0.06	1.40 ± 0.06	0.01 ± 0.005	200
104	98.55 ± 0.05	1.44 ± 0.04	0.02 ± 0.01	200
105	98.60 ± 0.04	1.38 ± 0.03	0.022 ± 0.014	200
106	98.45 ± 0.07	1.51 ± 0.05	0.033 ± 0.018	200
107	98.06 ± 0.18	1.91 ± 0.18	0.026 ± 0.014	400
108	97.86 ± 0.04	2.08 ± 0.03	0.06 ± 0.01	400
109	97.91 ± 0.02	2.04 ± 0.04	0.053 ± 0.014	400

(a) The first resin column loaded with clementine plutonium had a heel of 600 Mwd/ton material from the previous Hanford Metallurgical Waste Program.[2]

5.0 AMERICIUM CYCLE

5.1 Americium Recovery and Material Balance

The feed to the americium extraction column contained 2.505 g of americium and since 0.94 g was lost to the waste streams, 62.4 per cent of the americium should have been recovered. However, elution of the americium resin column at the end of the program yielded only 0.155 g of americium. Analysis of the eluted resin indicated that the resin retained 0.138 g. These values gave an exceedingly poor over-all material balance of 49.6 per cent. The samples of waste solutions,

particularly the IIAW samples, contained a great deal of precipitated salts, making it difficult to obtain a representative sample and to pipet an aliquot for analysis. The poor quality of the samples is probably the reason for the low material balance.

5.2 Feed Preparation

The americium recovery cycle was not operated when fuel elements containing 200 Mwd/ton plutonium were processed. The aqueous waste stream from the plutonium extraction column was neutralized with ammonia and sent directly to the waste storage tank (W-6). When 400 Mwd/ton material was processed, americium was recovered.

5.3 Americium Losses

The americium loss for the program was 37.6 per cent, based on analyses of collected waste solution. Individual stream losses are presented in Table 5-1.

Table 5-1
Americium Cycle Losses

Stream	Loss, % of IIAF Am
IIAW	14.8
IIBW	15.51
IIBPW	7.3
Total	37.6

6.0 PROGRAM COSTS

6.1 Total Cost

The total cost of the Clementine program was $56,834 (Table 6-1). A total of 14.7 kg of plutonium was recovered at a unit cost of $3.88 per gram.

6.2 Operating Cost

The Clementine program lasted from September 15 to October 15, 1954, and during this period, the operating cost was $42,608.

6.3 Postoperating Cost

The Metal Recovery Plant was shut down from October 15 to December 15, 1954 to convert the plant from plutonium recovery to irradiated uranium slug processing. The $77,958 cost for this period was prorated to the Hanford Metallurgical Waste and the Clementine Programs in proportion to the amount of plutonium processed in each program (67 kg during the Hanford program and 15 kg during the Clementine program). The Clementine program was charged $14,226 for postoperating costs.

Table 6-1

Summary of Metal Recovery Plant Costs for the Clementine Program

	Operating Cost		Program Cost		
Type of Expense	Subtotal Cost, $	% of Operating Cost	Total Cost, $	% of Total	Plutonium Cost, $/g
Operating Costs			42,608	75.0	2.91
Pilot plant labor	9,062	21.3			0.62
Analytical services	6,122	14.4			0.42
Chemicals and supplies	6,909	16.2			0.47
Protective clothing	975	2.3			0.07
Engineering and maintenance	1,049	2.5			0.07
Miscellaneous charges	362	0.8			0.02
Worked materials	4,674	11.0			0.32
Expense allocation	9,476	22.2			0.65
Health Physics	1,870	4.4			0.13
Research Director's Dept. charges	1,000	2.3			0.07
Division Director charges	1,109	2.6			0.07
Postoperating Cost			14,226	25.0	0.97
Total			56,834	100.0	3.88

7.0 REFERENCES

1. J. H. Hall letter to W. H. Lewis, TAD-1673 (Feb. 23, 1954).

2. R. E. Brooksbank, J. L. Matherne, W. R. Whitson, "Terminal Report on the Recovery of Plutonium and Americium from Hanford Metallurgical Waste in the ORNL Metal Recovery Plant," ORNL-1850 (Feb. 16, 1955).

3. D. C. Overholt, F. W. Foster, D. A. Orth, "An Ion Exchange Process for Plutonium Isolation and Purification" ORNL-1357 (Sept. 16, 1952).

4. R. H. Rainey, "Development of the Amex Process for Americium Recovery," ORNL-1697 (May 7, 1954).

8.0 APPENDIX

The dissolver data and the plutonium product analyses are summarized in Tables 8-1 and 8-2.

Table 8-1

Summary of Dissolver Data

Dissolving No.	Dissolver Data			Total Plutonium, g	
	Volume, liters	Plutonium α Activity, dts/min/ml	Specific α Activity, cts/min/mg	ORNL Analysis	Los Alamos S. F. Data
1	175	1.77×10^{8}	7.62×10^{9}	406.50	457.90
2	176	1.59×10^{8}	7.62×10^{9}	367.24	456.16
3	171	2.24×10^{8}	7.45×10^{9}	514.15	454.50
4	170	1.89×10^{8}	7.45×10^{9}	431.28	455.81
5	150	2.19×10^{8}	7.49×10^{9}	438.59	457.30
6	169	2.05×10^{8}	7.49×10^{9}	462.55	458.70
7	161	2.08×10^{8}	7.17×10^{9}	467.06	457.00
8	159	2.04×10^{8}	7.17×10^{9}	452.38	454.19
9	193	1.93×10^{8}	7.17×10^{9}	519.51	449.20
10	184	1.80×10^{8}	7.57×10^{9}	437.52	455.60
11	186	1.74×10^{8}	7.57×10^{9}	427.53	456.96
12	179	1.87×10^{8}	7.63×10^{9}	438.70	454.00
13	175	1.82×10^{8}	7.67×10^{9}	415.25	453.00
14	177	1.83×10^{8}	7.67×10^{9}	422.31	453.20
15	176	1.88×10^{8}	7.67×10^{9}	431.40	454.30
16	188	1.84×10^{8}	7.67×10^{9}	451.00	453.40
17	137	2.38×10^{8}	7.67×10^{9}	425.11	445.30
18	192	1.92×10^{8}	7.53×10^{9}	489.56	452.10
19	167	1.92×10^{8}	7.53×10^{9}	425.82	456.76
20	360	1.91×10^{8}	7.56×10^{9}	909.52	910.55
21	351	1.92×10^{8}	7.53×10^{9}	894.98	910.22
22	360	1.81×10^{8}	7.59×10^{9}	858.50	910.00
23	361	1.93×10^{8}	7.56×10^{9}	921.60	900.43
24	362	1.90×10^{8}	7.60×10^{9}	905.00	910.71
25	350	1.81×10^{8}	7.63×10^{9}	830.28	914.09
26	357	2.05×10^{8}	7.57×10^{9}	966.78	914.14
Heel	189	2.33×10^{6}	7.41×10^{9}	5.94	----
Total				14,716.06	15,005.52

Table 8-2

Summary of Plutonium Product Analyses

Product Container No.	Pu Conc, g/l	Total Pu, g	H^+, N	Gross α, cts/min/ml	Specific α, Activity, cts/min/mg	U, g	U in Pu, %
N-47	49.18	881.50	4.10	3.75×10^9	7.62×10^7	2.474	0.28
N-23	51.81	900.46	4.40	3.86×10^9	7.45×10^7	3.580	0.40
N-20	54.72	957.11	4.20	4.10×10^9	7.49×10^7	7.294	0.76
N-9	67.60	1,237.82	4.20	4.85×10^9	7.17×10^7	3.003	0.24
N-19	48.32	851.50	3.60	3.66×10^9	7.57×10^7	7.014	0.82
N-29	42.45	753.19	3.20	3.24×10^9	7.63×10^7	5.678	0.75
N-21	56.46	1,004.99	3.80	4.33×10^9	7.67×10^7	7.049	0.70
N-46	50.34	913.42	4.10	3.86×10^9	7.67×10^7	8.111	0.89
N-31	56.59	990.44	4.60	4.26×10^9	7.53×10^7	9.241	0.93
N-38	52.75	913.68	4.50	3.99×10^9	7.56×10^7	5.456	0.60
N-30	47.04	831.53	4.60	3.54×10^9	7.53×10^7	6.169	0.74
N-40	59.81	1,115.93	4.00	4.54×10^9	7.59×10^7	10.337	0.93
N-24	50.63	867.14	4.10	3.83×10^9	7.56×10^7	4.624	0.53
N-15	42.65	777.25	4.20	3.24×10^9	7.60×10^7	2.570	0.33
N-17	50.49	916.90	4.05	3.85×10^9	7.63×10^7	1.780	0.19
N-25	32.62	604.71	4.70	2.47×10^9	7.57×10^7	6.803	1.12
N-32	3.28	67.93	5.50	2.18×10^8	6.65×10^7	9.506	14.00
N-48	9.30	181.09	4.30	6.89×10^8	7.41×10^7	5.647	3.12