





CRITICALITY EXPERIMENTS WITH MIXED PLUTONIUM AND URANIUM NITRATE SOLUTION AT A PLUTONIUM FRACTION OF 0.5 IN SLAB AND CYLINDRICAL GEOMETRY

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SUMMARY

A series of critical experiments was completed with mixed plutonium-uranium solutions having Pu/(Pu + U) ratios of approximately 0.5. These experiments were a part of the Criticality Data Development Program between the United States Department of Energy (USDOE), and the Power Reactor and Nuclear Fuel . Development Corporation (PNC) of Japan. A complete description of, and data from, the experiments are included in this report. The experiments were performed with mixed plutonium-uranium solutions in cylindrical and slab geometries and included measurements with a water reflector, a concrete reflector, and without an added reflector. The concentration was varied from 112 to 332 g (Pu + U)/liter. The ratio of plutonium to total heavy metal (plutonium plus uranium) was 52% for all experiments.

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CRITICALITY EXPERIMENTS WITH MIXED PLUTONIUM AND URANIUM NITRATE SOLUTION AT A PLUTONIUM FRACTION OF 0.5 IN SLAB AND CYLINDRICAL GEOMETRY

1.0 INTRODUCTION

The design and operation of facilities for recycling fast breeder reactor fuels involves criticality conditions which are much different from those encountered in the light water reactor fuel cycle. Conditions are encountered in plant operations with fissionable materials that involve complex equipment shapes, high plutonium content in solution with uranium, and neutron absorbing materials that affect criticality. Experimental criticality data are required for validation of the calculations and nuclear data used in facility design, operational procedures and related licensing activities to ensure freedom from criticality accidents. In August, 1983 the U. S. Department of Energy (DOE) and the Power Reactor and Nuclear Fuel Development Corporation (PNC) of Japan entered into an agreement to study criticality aspects of nuclear fuels encountered in the development of fast breeder reactor recycle technology. This arrangement was developed through the DOE and PNC Agreement in the Field of Liquid Metal-Cooled Fast Breeder Reactors. Prior to this Joint Memorandum of Agreement (MOA) for Nuclear Criticality Data Development Programs, DOE had initiated an experimental program at the DOE Hanford Critical Mass Laboratory to provide basic criticality data on plutonium-uranium systems in support of the U. S. Liquid Metal Fast Breeder Reactor Program. Under this MOA, PNC has promoted and enlarged the DOE Program to cover areas of mutual interest as well as areas of specific interest to PNC.

Some computer codes for criticality calculations have been developed and applied to FBR fuel cycle facility designs. Application of these codes, however, and the associated cross-section libraries, results in uncertainties in the criticality aspects for FBR fuel under the conditions encountered. Therefore, experimental data are needed which will permit validation of codes and cross-section data to minimize the uncertainties so that facility safety, efficiency, and reliability can be enhanced. The verification of criticality evaluation methods is the subject of regulatory licensing activity.

This report contains a description of, and data from, the criticality experiments conducted with mixed plutonium-uranium solutions at Pu/(Pu + U)ratios of approximately 0.5, which documents part of the work in the Project's Subtask 120. The experiments were performed in cylindrical and slab geometry. Reflector conditions of water reflected, concrete reflected and bare were used. The solution concentration was varied from 112 to 332 g (Pu + U)/liter. These data have application whenever mixtures of plutonium and uranium exist, in the head-end of a fuel reprocessing plant through the first solvent extraction cycle, in storage vessels and during product conversion when a coprocessing scheme is used.

2.0 DESCRIPTION OF EXPERIMENTAL ASSEMBLIES

This section includes the general description of the experimental assemblies used for obtaining the criticality data.

2.1 GENERAL DESCRIPTION OF THE SOLUTION SYSTEM

An existing experimental system, previously used for solution experiments at the Critical Mass Laboratory, was used in the measurements to provide the data for this report. The solution system is located in the critical assembly room. The addition of solution to the experimental vessel is remotely made from the control room. The layout of equipment in the critical assembly room is shown in Figure 2.1.

The critical assembly room is 10.67 meters square and has a ceiling height of 6.4 meters. The side walls are composed of 1.52 meter thick concrete. The concrete ceiling and floor are each 0.61 meters thick.

The containment hood (Hood 1) was located 1.83 meters from the north wall of the room. The west side of the hood, which faces the wall containing the DS and DM tanks was located 1.52 meters from that wall.

A schematic showing the piping connections between the three experimental vessels is shown in Figure 2.2. This piping arrangement allows critical experiments to be conducted with the same solution in each of three vessels without changing vessels. The small diameter cylinder (35.39 cm) and the variable thickness slab tank were used in these series of experiments.



Figure 2.1 Floor Schematic Plan of the Critical Assembly Room

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Figure 2.2 Piping Schematic for the Three Experimental Vessels

2.2 CYLINDRICAL VESSEL ASSEMBLY

A photograph of the cylindrical vessel system is shown in Figure 2.3. This system contains two cylindrical vessels. The vessel used for the experiments in this report has an inside diameter of 35.39 cm. This vessel can be seen through the window on the left. The control and safety blade mechanisms are mounted above the vessel and can be seen in the figure directly above the vessel. The reflector tank serves to contain water when water reflected vessels are used. Windows of polycarbonate (Lexan) were installed on the fronc for access to the tank and for visual inspection. This reflector tank was fabricated of carbon steel. The placement of the cylindrical vessels is shown in Figure 2.4.

The small cylindrical vessel (35.39 cm ID and 106.60 cm inside height) was fabricated of 304L stainless steel. The wall thickness was 0.079 cm. The control and safety blades are external to the vessel and are fully withdrawn during the neutron flux determination during the critical approach measurement. A schematic of the cylindrical vessel is shown in Figure 2.5.

The fill, dump and manometer lines enter the bottom of the vessel through the dump valve system. The vessel is connected to the dump valve pedestal by a Marmon flange connection which provides a leak tight seal.

The experiments with the cylinder were conducted with the reflector tank empty, with the reflector tank containing water and with a concrete reflector positioned around the cylindrical vessel. In the "bare" condition, the reflector tank is empty, but some neutrons will, however, be reflected from the adjacent tank walls and the large empty cylinder that is also located in the reflector tank, and the concrete walls of the room. Reflector type control and safety blades of acrylic resin were used for the bare assembly. For the water reflected cases, the reflector tank was filled to a level slightly below the top of the cylindrical vessel.



Figure 2.3 Photograph of the Cylindrical Vessel System

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In the case of the concrete reflected assemblies, the concrete was 25.2 cm thick. An artist's conception of the concrete reflector, showing the concrete positioned around the experimental vessel, is shown in Figure 2.6. (See Appendix A for Engineering Drawing). The concrete reflector extended to the bottom of the reflector tank.

Engineering drawings are provided in Appendix A for the cylindrical vessel system; these contain detailed dimensions used for fabrication.

2.3 EXPANDABLE SLAB TANK SYSTEM

The expandable slab tank system has been in use for several years and has been used in conducting many experiments (Lloyd, 1973). The engineering drawings for this system are provided in Appendix B. C. R. Richey has performed an analysis on the grid structure for the slab tank system (Richey, 1967) in which he determines its effects on the experiments.

The slab assembly (Figure 2.7) used in these experiments is unique because its thickness can be adjusted over a range of 7.6 to 22.8 cm. This range of adjustments is made possible by means of a stainless steel bellows fabricated around the periphery of the tank. Slab thickness change is accomplished by means of adjustment screws, located at the corners of the vessel, between the opposite sides of the slab; slab thickness is measured by means of a dial caliper. The height and width of the tank is about 106.7 cm, based on the average of the bellows variation. The stainless steel sides (0.159 cm) are reinforced with an egg-crate-type structure that maintains the side position to within ~0.025 cm when filling the tank. The assembly and its parts are of Type 304L stainless steel. A reflected assembly is achieved by attaching gasketed side plates and filling with water. The assembly is positioned in a large hood for contamination control, in the event of leaks.



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The grid structure is composed of 0.3175 cm thick stainless steel plate. The measured average center-to-center spacing of the support grid structure is 10.6 cm. The bellows, visible in Figure 2.7, is constructed with a tip to trough distance of 5.08 cm at a slab thickness of 15.2 cm. The bellows is composed of two sets of four V-shaped structures; the sets are separated by a U-shaped structure having a width of 2.86 cm and a depth of approximately 2.5 cm. The corners of the slab tank are rounded with a 14.4 cm radius measured from the outside of the tank. The bellows are made of type 304L stainless steel and are 0.079 cm in thickness.

The slab tank is positioned inside a metal frame (visible in Figure 2.8) to which gasketed side plates can be attached to form a reflector tank. In both water reflected and "bare" experiments these side plates are present except where noted. In case a water reflector is used, the water level is set at the midpoint of the top bellows of the slab tank. The side plates and end plates for the reflector tank are 0.635 and 0.476 cm thick respectively and are fabricated from stainless steel. A schematic of the expandable slab tank positioned in the reflector tank is shown in Figure 2.9.

The inside dimensions of the water reflector tank are 68.6 cm wide by 142.2 cm long by 143.5 cm high. The slab tank is positioned such that the center of the bellows on the lower edge of the slab is 18.4 cm above the inside face of the lower plate. The slab tank is positioned such that the narrow side faces (Bellows Center) are 17.75 cm from the inside face of the water reflector tank. The distances from the broad faces of the slab tank to the water reflector tank walls is varied according to the critical configuration being examined, from 25.6 cm to 33.2 cm on the south side; and 20.2 cm to 27.8 cm on the north side for tank thickness settings of 22.8 cm to 7.6 cm, respectively.

The slab tank and associated water reflector tank are surrounded by a containment hood (see Figure 2.10). The hood has a stainless steel structure with Plexiglas windows which are 0.95 cm thick. The steel structural material is approximately 0.635 cm thick.



Figure 2.8 Critical Experiments Expandable Slab Assembly



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Figure 2.10 Containment Hood for Expandable Slab Assembly (Looking at South Side of Slab)

The bottom of the water reflector tank is located 100.6 cm above the concrete floor of the experimental cell. The broad faces of the water reflector tank are located 34.3 and 138.4 cm, from the South and North faces of the hood walls, respectively. The narrow faces of the reflector tank are located 24.6 and 109.5 cm, from the West and East hood walls, respectively.

The slab tank is equipped with control and safety blade drive mechanisms which provide for insertion of the control and safety blades directly into the solution. The control and safety rod drive mechanisms are mounted above the slab tank. A 2.5 cm thick steel grate platform is mounted inside the hood, 230 cm above the floor. The grate provides a working platform for the installation of the control and safey blade drive mechanisms.

3.0 EXPERIMENTAL RESULTS

This section provides the results of the experiments including a description of the measurement techniques involved in obtaining the data.

3.1 CRITICALITY MEASUREMENT TECHNIQUES

The critical heights for the experiments reported herein were determined using the critical approach method (Clayton, 1985). In this critical approach method, neutron flux measurements are made as the height of solution is incrementally increased. Inverse count rate is plotted versus solution height. As the delayed critical condition is approached the neutron count rate approaches infinity so the inverse count rate approaches zero. By extrapolation of the inverse multiplication curves to zero value, the critical height is determined for the system. The neutron flux is routinely taken on three boron-lined proportional detectors located near the experimental vessel. The data from the three counters extrapolate to essentially identical values for critical height at near critical values. The computer calculated least squares fit of the inverse multiplication curves, used in determining the critical value of the heights for each experimental assembly, are included in Appendix C. The critical heights given in Appendix C for the slab tank are with respect to the top of the bottom bellows.

3.2 CRITICALITY DATA

The criticality data for this report were obtained from January - September 1985, when three experiments were completed with the small cylindrical vessel and seven with the expandable slab experimental system. The data are summarized in Tables 3.1 and 3.2. The critical indicated heights for the slab tank are shown with respect to the midpoint of the lower bellows. The chemical analyses data for the (Pu + U) nitrate solutions given in these tables were provided by the Chemical and Analysis Section of the Westinghouse Hanford Co. from samples of solution supplied to them. The sample analyses methods and descriptive titles are given in Table 3.3. The critical heights were calculated by a least squares

fit to the inverse neutron multiplication data from three neutron detectors. (Computer printout provided in Appendix C). The 241 Am content for each sample analyzed, the analysis date and the experiments covered by that sample are given in Table 3.4. The isotopic analyses values for the plutonium and uranium of the experiments are given in Table 3.5. Table 3.6 provides information on the temperatures of the critical assembly room (CAR), the dump mix tank (DM) and the water reflector. Also in Table 3.6, the reflector water level and the position of the bottom of the control and safety blades are given. (Reference is the vessel top).

Appendix D provides data on the chemical analyses for the impurities found in the (Pu + U) nitrate solutions.

The chemical analyses of the reflector water samples are given in Appendix E.

The composition of the concrete reflector is given in Appendix F. Also provided are the calculated atomic densities used in previous measurements using this concrete reflector (Primm, 1986).

Run Date	Project Case Number	CML Experiment Number	Reflector	Sample ⁻¹⁾ Number	Pu (g/liter)	U (g/liter)	Specific Gravity	Free Acid H⁺	Critical Height, H _c (cm)
01/15/85	1	Ø46	Water	1087	59.35	53.12	1.1968	Ø.77	23.83
01/17/85	1	Ø46R	Water	1087	59.35	53.12	1.1968	Ø.77	24.06
01/29/85	2	647	Concrete	1088	59.82	54.12	1.1977	0.75	24.88
04/26/85	10	Ø 51	Bare	1096	59.63	53.27	1.1978	Ø.85	34.93

$\frac{\text{TABLE 3.1}}{\text{Solution in 35.39 cm I. D. Cylinder}}$

-1) Samples were not ion exchanged prior to analysis

TABLE 3.2 Criticality Measurements with (Pu + U) Nitrate Solution in Slab Geometry

Run Date	Project Case Number	CML Experiment Number	Reflector	Sample ⁻³⁾ Number	Pu (g/liter)	U (g/liter)	Specific Gravity	Free Acid H⁺	Slab Thickness cm	Solution Critical Height, cm
Ø4/Ø5/85	3	Ø48	Bare -1) (1 Side Tamper Tank)	1Ø95A	174.07	157.48	1.5316	1.15	17.46	*
Ø4/Ø8/85	3	Ø49A	Bare -1) (1 Side Tamper Tank)	1 0 95 a	174.07	157.48	1.5316	1.15	18.10	78.74
04/10/85	3	050	Bare -2)	10958	174.62	157.52	1.5329	1.15	18.10	71.88
09/05/85	6	Ø54	Water	1119	118.94	108.39	1.3714	Ø.82	12.19	60.62
Ø9/Ø6/85	7	Ø55	Water	1120	60.53	55.17	1.1944	0.55	12.19	83.41
Ø9/Ø9/85	8	Ø56	Bare -2)	1121	61.00	55.52	1.1954	Ø.53	19.05	45.31
Ø9/1 Ø/85	8	Ø56a	Bare -2)	1121	61.00	55.52	1.1954	Ø.53	17.78	60.15

*Data show criticality not possible in the slab with 17.46 cm thickness with Pu + U nitrate solution being used in this experiment.

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-1) The north side was removed

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-2) Both sides of tamper tank on

-3) Samples were not ion exchanged prior to analysis

TABLE 3.3 Chemical Analyses Methods

<u>Measurement</u>	Method Title $^{-1}$		Date of <u>Approval</u>
Plutonium	Plutonium by Automated Amperometric Titration.	(30.3)	03/18/85
Uranium	Uranium by Automated Potentiometric Titration.	(30.8)	02/05/86
Impurities	Impurities by Emission Spectroscopy: Direct Reader.	(40.13)	06/11/85
241 _{Am}	Americium-241 by Anion Exchange and Alpha Analysis.	(40.16)	05/14/75
Free Acid	Determination of Free Acid in Uraniu Plutonium Solutions. (Using an improved oxalate method)	m/ (40.22)	02/04/86
Density	Density of Solutions. (Using Mettle Paar Density Meter)	r/ (40.23)	02/05/86
Isotopic	Isotopic Composition of Plutonium and Uranium by Mass Spectroscopy.	(30.6)	09/27/78
Impurities	Impurities by Spark Source Mass Spectrometer.	(40.15)	05/22/75

-1) The numbers in brackets are HEDL's method numbers.

Sample Number	241 _{Am} (ug/ml)	Analysis Date
1087	290	01/29/85
1095	921	10/21/85
1096	306	05/08/85
1096	307	05/08/85
1119	587	09/23/85
1121	302	09/23/85

TABLE 3.4Chemical Analysis Values for Americium-241in Micrograms per milli-liter

Sample 1087 covers experiments 046, 046R and 047

Sample 1095 covers experiments 048, 049A and 050

Sample 1096 covers experiment 051

Sample 1119 covers experiment 054

Sample 1121 covers experiments 055, 056 and 056a.

TABLE 3.5	Isotopic	Analyses	Values	of	Pu	and	U	(Wt%))
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	<u>Sample 1087-1)</u>	<u>Sample 1095⁻²⁾</u>	<u>Sample 1119⁻³⁾</u>	Sample 1121 ⁻⁴⁾
<u>Pu</u>				
238	0.029 ± 0.003	0.021 ± 0.002	0.029 ± 0.001	0.027 ± 0.001
239	91.11 ± 0.04	91.11 ± 0.04	91.10 ± 0.04	91.10 ± 0.04
240	8.30 ± 0.04	8.31 ± 0.04	8.31 ± 0.04	8.31 ± 0.04
241	0.474 ± 0.002	0.462 ± 0.003	0.464 ± 0.002	0.467 ± 0.002
242	0.093 ± 0.002	0.092 ± 0.002	0.093 ± 0.002	0.092 ± 0.002
<u>U</u>				
238	99.253 ± 0.005	99.267 ± 0.005	99.265 ± 0.006	99.266 ± 0.006
236	0.027 ± 0.002	0.022 ± 0.002	0.022 ± 0.001	0.022 ± 0.001
235	0.707 ± 0.005	0.701 ± 0.004	0.705 ± 0.005	0.702 ± 0.005
234	0.012 ± 0.001	0.009 ± 0.002	0.009 ± 0.003	0.009 ± 0.003

-1) Sample 1087 is for Experiments 46, 46R, 47 and 51: Date analyzed, 1/31/85.
-2) Sample 1095 is for Experiments 48, 49 and 50: Date analyzed, 10/24/85.
-3) Sample 1119 is for Experiment 54: Date analyzed, 9/25/85.
-4) Sample 1121 is for Experiments 55, 56 and 56a: Date analyzed, 9/25/85.

			. .	-0	Reflector Level	Control and Safety Blade Distance
	Experiment		Temperature	<u>c</u> •	Distance Below	Below Vessel
	Number	Koom	Storage lank	Reflector	Vessel lop (cm)	lop (cm)
Cylinder	Ø46	22.8	17.8	17.6	1.27	2.54
	Ø46R	22. 🛙	17.7	20.3	1.27	2.54
	647	21.9	17.9	21.9	N/A	2.54
	Ø51	17.1	18.6	N/A	N/A	1)
Slab	Ø48	22.0	18.9	N/A	N/A	5
	Ø49A	21.0	17.0	N/A	N/A	5
	050	19.0	20.0	N/A	N/A	5
	Ø54	22.2	24.6	22.5	2.54	5
	055	23.5	24.7	22.2	2.54	5
	Ø58	22.7	24.9	N/A	N/A	5
	Ø56a	21.1	24.6	N/A	N/A	5

TABLE 3.6 Information on Temperatures, Reflector Level, and Control and Safety Blade Position

N/A - Not Applicable

1) Control & Safety blade top was 1.905 cm below tank bottom.

3.3 SOURCES OF ERROR

It is practically impossible to assess, individually, the effects of all the uncertainties in all of the experimental measurements. Realistically, it is only necessary to examine those variables or combination of variables which might have a reactivity effect which is a significant fraction of the typical uncertainty in a particular KENO calculation. This evaluation was done for the significant measurements involved in earlier experiments and reported (Primm, 1986). From that analysis it was found that the primary uncertainty that caused significant error was from the free acid values. Since those measurements, a study was made and a free acid analysis method developed and reported (J. L. Ryan, 1985). This has significantly reduced uncertainties in the analysis for free acid. Further work provided free acid standards so that the analyses could be confirmed.

The evaluation of uncertainties by (Primm, 1986) included the critical height, plutonium concentration, uranium concentration, density, free acid and composition of reflectors. It was recognized by (Primm, 1986) that the procedure used to derive uncertainties due to experimental and chemical analysis measurements were likely to over estimate the value of each parameter.

The latest estimated values of uncertainties are listed in the following table:

TABLE 3.7 Measurement of Uncertainties

Pu Concentration ± 0.1% U Concentration ± 0.1% Specific Gravity ± 0.0003 Free Acid ± 0.04 M Critical Height ± 1.6 mm Values for uncertainties in the chemical analyses were provided by M. C. Burt of the Chemical and Analysis Section of the Westinghouse Hanford Company, Hanford Engineering Development Laboratory. The critical height uncertainty is given as 1.6 mm though the least square fitting of approach data for three counting systems would indicate a smaller value as reasonable. The 1.6 mm is the smallest unit on the sight tube.

4.0 REFERENCES

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5.0 ACKNOWLEDGEMENTS

The work performed for this report required the cooperation and assistance of a number of people, some of whom are listed below. Their contributions are greatly appreciated.

- o K. H. Rising (DOE-RL) for assistance in administrative matters.
- o E. D. Clayton for information and guidance on technical matters.
- o M. C. Burt for providing accurate chemical analyses of solutions in a timely manner.
- o J. H. Smith as Senior Reactor Operator in providing valuable advise, and assistance in performing the experiments.
- o L. N. Terry for typing, proofreading and guidance in preparation of this report.

APPENDIX A

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ENGINEERING DRAWINGS OF THE CYLINDRICAL VESSEL SYSTEM

APPENDIX B

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ENGINEERING DRAWINGS OF THE EXPANDABLE SLAB TANK SYSTEM

APPENDIX C

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LEAST SQUARE FITS OF THE CRITICAL APPROACH DATA

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APPENDIX C

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LEAST SQUARE FITS OF THE CRITICAL APPROACH DATA

The value of the extrapolation for the expandable slab system requires the addition of one inch (2.54 cm). This is due to the zero position of the solution height measuring system having to be at the top of the bellows. The solution heights on all least square plots are in inches.



CFRP-PNC-2-14-2-048 DATE 1-14-85 14" CYL. H20 REFL. CB&SB OUT

Figure C.1 Least Square Fit CFRP-PNC 046



Figure C.2 Least Square Fit CFRP-PNC 046R

C.3



Figure C.3 Least Square Fit CFRP-PNC 047



CFRP-PNC-3-7.25-1-049A DATE 4-8-85 7.125" INSIDE WIDTH SLAB TANK NO REFL. NORTH T.T. SIDE OFF. SAMPLE#1095A CB&SB OUT BACK-OFF



C.5



CFRP-PNC-3-7.25-1-050 DATE 4-10-05 7.125" INSIDE WIDTH SLAB TANK ALL T.T.SIDES ON. NO REFL. SMPLE #10958 CB&SB OUT

Figure C.5 Least Square Fit CFRP-PNC 050



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Figure C.6 Least Square Fit CFRP-PNC 051



CFRP-PNC-3-4.925-2-054 DATE 9-5-85 SLAB TANK

Figure C.7 Least Square Fit CFRP-PNC 054



CFRP-PNC -3-4.925-2-055 DATE 9-8-85 SLAB TANK CB & SB OUT. SAMPLE# 1120 ZER0=0.500"

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Figure C.8 Least Square Fit CFRP-PNC 055



Figure C.9 Least Square Fit CFRP-PNC 056



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Figure C.10 Least Square Fit CFRP-PNC 056A

CHEMICAL ANALYSES DATA OF THE IMPURITIES IN (Pu + U) NITRATE SOLUTIONS

APPENDIX D

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APPENDIX D

CHEMICAL ANALYSES DATA OF THE IMPURITIES IN (Pu + U) NITRATE SOLUTIONS

The chemical analyses data for sample 1097 are for experiments 046R, 047 and 051. The analyses data for 1095 are for experiments 048, 049A and 050. The analyses data for 1119 are for experiment 054. The analyses data for 1121 are for experiments 055, 056 and 056a.

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Figure D.1 Spectrographic Analysis Report # 1087

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Figure D.2 Spectrographic Analysis Report # 1095

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Mg	200	300	Y			Dy	
AI	2000	2000	Zr			Но	
Si	200	2000	Nb			Er	
Р	30	30	Мо	2	2	Tm	
S	200	300	Ru			Yb	
CI	10	100	Rh			Lu	
к	100	300	Pd			Hf	
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Figure D.3 Spectrographic Analysis Report # 1119 and 1121

APPENDIX E

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CHEMICAL ANALYSES DATA OF THE REFLECTOR WATER SAMPLES

TABLE E.1 Water Sample Analysis - 046 and 046R



HANFORD ENVIRONMENTAL HEALTH FOUNDATION

April 2, 1985

9475

Pacific Northwest Laboratory 209-E Building 200-E Area

Attn: Ray Lloyd

WATER SAMPLE ANALYSIS - TAMPER 046

One water sample, Tamper 046, has been analyzed for the parameters requested. Analysis was done in accordance with <u>Standard Methods for Water and</u> <u>Wastewater</u>, 15th ed.

Parameter	(mg/L) Tamper 046
рH	7.0
Total alkalinity	56.9
HCO ₃ alkalinity	54
CO ₃ alkalinity	<0.5
Total Dissolved Solids	105
Sulfate	3.8
Nitrate as N	0.10
Fluoride	0.12
Chloride	2.7
Zinc	0.13
Manganese	0.02
Lead	< 0.005
Copper	<0.01
Chromium	<0.01
Iron	0.14
Cadmium	<0.005

If you have any questions, please contact Environmental Health Sciences.

Maurent. Hamilton

M. K. Hamilton, CIH Laboratory Director Environmental Health Sciences

spm

66282 O. 8 OX 100. RICHLAND, WASHINGTON 99352

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HANFORD ENVIRONMENTAL HEALTH FOUNDATION

November 19, 1985

CO 10165

Pacific Northwest Laboratories 209-E Building, 200-E Area

Attn: R. Lloyd

WATER ANALYSIS

Following are the results of the water analysis of two samples received October 22. All analyses were performed in accordance with <u>Standard Methods for the Examination of Water & Wastewater</u>, 16th Ed.

Analysis	054 <u>9-5-85</u>	05 5 10-21-85
рН	7.3	7.3
Total alkalinity mg/L as CaCO ₃	49.1	46.6
Bicarbonate alkalinity mg/L as CaCO3	47	44
Carbonate alkalinity mg/L as CaCO3	<0.5	<0.5
Total dissolved solids mg/L	77	92
Sulfate mg/L	15	15
Nitrate (as N) mg/L	<0.1	<0.1
Fluoride mg/L	0.12	<0.1
Chloride mg/L	0.43	0.45
Cadmium mg/L	<0.0002	0.0007
Copper mg/L	0.02	<0.01
Chromium mg/L	<0.01	<0.01
Iron mg/L	0.08	1.49
Lead mg/L	<0.002	0.006
Manganese mg/L	<0.01	0.025
Zinc mg/L	<0.05	4.2

If there are any questions concerning this analysis, please contact us.

Mauricin K. Hamilton

M. K. Hamilton, CIH Laboratory Director Environmental Health Sciences

P. O. BOX 100. RICHLAND. WASHINGTON 99352

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APPENDIX F

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COMPOSITION OF CONCRETE REFLECTOR

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APPENDIX F

COMPOSITION OF CONCRETE REFLECTOR⁻¹⁾

Two samples of the concrete reflector were analyzed for material composition by the use of two techniques - x-ray fluorescence (XRF) and isotope neutron activation analysis (INAA). The procedure involved obtaining a section of the center of the core and reducing the material to powder such that it would pass through a 140 mesh screen. Two aliquots were obtained. Each aliquot was analyzed by XRF and INAA).

The INAA procedure involved a 5-minute irradiation in the neutron multiplier facility, a 5-minute delay, and then a counting of 600 seconds to obtain Al, V, Ti, Mg, and Ca. Approximately 2 hours later the samples were recounted for 1000 seconds to obtain data for Na, K, and Mn. A summary of this data is found in Table F.1.

The XRF procedure involved pelletizing the material to form a thin wafer. The samples were then analyzed by both a Zr and then a Ag secondary source. These data are also shown in Table F.1. The reported error is the 1σ value for each sample and the weighted standard deviation for the mean value $[1/\Sigma(1/\sigma_1^2)]$.

Each sample was then separated into two aliquots. The water content was determined by the following procedure. Each of the samples was weighed, then heated to 100° for 1 hours, cooled, and reweighed. The samples were then heated to 1000° for 1 hour, cooled, and weighed. The results of the analyses are shown in Table F.2. Based on the data in Table F.2, the hydrogen content of the concrete is determined to be 1.05 ± 0.03 weight percent.

⁻¹⁾ Analyses performed by Elwood Lepel, Pacific Northwest Laboratory documented in a memorandum to Mike Durst, PNL, dated May 27, 1981.

	Unit of		TNA	-b)_		XRF			
	Measure		1NA/	2		1	2	Ave	erage
Element	(WE)								
N-	•	1.40	+ 0.01	1.46	± 0.01	-	-	1.43	± 0.00/
Na	70 97	0.84	+ 0.46	1.00	± 0.50	-		0.92	± 0.34
мg		5 27	+ 0.03	5.58	+ 0.03	4.0 ± 1.4	4.3 ± 1.4	4./9	± 0.02
AI	70 07	J•21	<u> </u>	210-	-	22.3 ± 1.5	23.9 ± 1.6	23.1	± 1.1
51	70		_		-	<0.6	<0.6		
Ρ	76		-		-	0.40 ± 0.09	0.36 ± 0.09	0.38	± 0.06
5	76	0.96	- 0.14	0 70	+ 0.15	0.67 ± 0.04	0.67 ± 0.04	0.72	± 0.3
К	*	0.80	± 0.14	10.70	+ 0.5	13.8 ± 0.7	12.9 ± 0.7	12.0	± 0.3
Ca	76	10.8	± 0.5	2000	+ 200	2900 ± 200	2700 ± 200	3320	± 122
TI	ppm	3910	± 340	2900	± 200	2300 - 200 01 + 31	140 ± 30	102	± 3
V	ppm	91.3	± 3.0	05.5	1 5.0	106 + 21	134 ± 19	165	± 14
Cr	ppm	•	-		- 	130 - 21 606 + 36	577 ± 34	565	± 3
Mn	ppm	557	± 4	520	I 4	0.17 +	3.29 ± 0.16	3.37	± 0.12
Fe	%	•	-		- 3.45	0.17 ±	<49	<49	
Со	ppm	•	-		-	~49 r7 A	52 + 6	54	± 3
N1	ppm		-		-	5/ ± 4	30 + 3	40	+ 2
Cu	ppm		-		-	42 ± 5	146 ± 8	161	+ 6
Zn	DDW		-		-	$1/6 \pm 9$		11 5	+ 0.7
Ga	ppm		-		-	12 ± 1		28	+ 1
Pb	DDM		-		-	34 ± 2		20	+ 1
As	DDM		-		-	33 ± 2	29 ± 2	20 E	+ 07
Rb	DDM		-		-	21 ± 1	20 ± 1	20.5	± 0.7
Sr	000		-		-	280 ± 20	300 ± 30	290	÷ 1/
Ŷ	DDm		-		-	15 ± 1	15 ± 1	12	± 0./
7r	DD m		-		-	85 ± 7	87 ± 7	80	± 5
-1 Nb	PP"		-		-	5.4 ± 1.0	5.3 ± 0.7	5.4	± 0.0
Mo	PPm		-		-	6.9 ± 0.8	5.6 ± 0.8	0.2	± U.0
nu	P. P								

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<u>TABLE F.1</u> Concentration of Elements in Concrete Reflector -a)

(a) Reported error is the $l\sigma$ value for each sample. The weighted standard deviation is reported for the average.

(b) Isotope neutron activation analysis method.(c) X-ray fluorescence method.

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Sample Number	Weight Loss After Heating to 100 ⁰ C (wt%) ⁻ a)	Weight Loss After Heating to 1000 ⁰ C (wt%) ^{-a)}
A1	3.0	9.2
A2	2.9	9.2
B3	3.1	9.6
B4	3.3	9.8
Average	3.1 ± 0.2	9.4 ± 0.3

TABLE F.2 Water Content of Concrete Reflector

(a) Referenced to original sample weight.

The major constituents of the concrete are listed in Table F.3. The unidentified mass from the analysis was assumed to be oxygen. The density of the concrete was determined to be 2.3 \pm 0.1 g/cm³. The computed atom densities for each element are also listed in Table F.3.

Element	Weight Percent	Atom Density (Atoms/b°cm)
0	51.91	4.553E-2
Si	23.10	1.154E-2
Ca	12.00	4.201E-3
۲A	4.79	2.491E-3
Fe	3.37	8.468E-4
Na	1.43	8.728E-4
н	1.05	1.462E-2
Mg	0.92	5.310E-4
κ	0.72	2.584E-4
S	0.38	1.663E-4
T1	0.33	9.667E-5

<u>TABLE F.3</u> Calculated Atom Densities for the Concrete Reflector

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PNL-5768

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4	646[MATCA		PRIMER ALEYD TYPE A	IND 2 COATS SURIALD	SS EMAMEL COLON TO DE	1	1 14	SOLENOID CAT	88 CA 8586 8 18	A1A 1	EL REPAL
	NC 8460W		SELECTED BY CUST.				_	SIDE MOUNTED	230 VAC		10001010
10	INSIDE DIAMETER	•.	PIPE END PREPARATIO	IN WHERE REQUIRED S	HALL HAVE 37 1/2" BEVEL	1		ENIFE GATE DA	LVE CAT NO 0300	METAL	BE ZURSE
	E1P (10007)		· · · · · · · · · · · · · · · · · · ·	·				STATES BOUND	POBT 384 517 AC	TUATOR CAT	
1	LONG	10.	BINERSIDES AND JOLE	BANCES ARE IN ACCO	ADABCE WITH ARSE TI4.5.			SUPPLY	0/// 20-100 /21		
MATL NAT	MAS18UM	l ".	TOLERANCES FOR PH	18			1.	FEMALE COMMER	TOR CAT NA 11-1	708.7.12	
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APT	AMERICAN STANDARD TAPER THREADS		BECSAAL 2.0 ARSUIAR	/3 ⁴		~H-	+-	MALE CONVECTO			
85	NEAN SIDE	1	1/				+-"	union (1804	LAI 88 53-400-8	,	
	WWISIDE DIAMETER	l ".		1. 7. 7. 7. 7 <u>8. 1</u> . 18. 		Ľ	1"	3/4" #E1460#	NIAD PLUE		1 311 351
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151	POUNES PER SQUARE ENCH		ANGULAR . 1/	/ 1 •		1	1	0-8185 CAT 80	42-394 (¥1708-	A)	PARALE
11	LIQUID PLACTHART ELAMINATION	· ·	TOLERANCES FAR PR I	2. 3. 9. 10 4 11		T	1	0-8186 CAT 89	#2-381 (VITOR-	A)	PARLES
QTY.	Quantity		FRACTIONAL 1/1	J		-	1	6456ET 1/8" T	HICK # 7 1/2* 6		TEFLON
	RADIUS	Δ ¹⁴ .	THE FOLLOWING PART		LACED BY THE ARBULAR			HOLES TO MATC	H 78 9		
86-848	REINFORCING BAA	_	TE ASSEMBLT (H-2-9	\$7413. 5. 4. 9(1).	10, 11, 12, 17(1).	•	11	6ASEET 1/8" 1	HICE E 7 1/2* 6	SO 2 3 1/2	TEFLOR
	BEFERINCE	1	19(1), 23(1), 24(1)), JO(12), J1(6), A	40 38(6).			10 HOLES TO M	ATCH FLABSE		
8000		ł				1	1	1/8" SASLET P	08 3" 150 <i>4 F</i> LAN	16E	TEFLON
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