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Criticality Experiments with Mixed Plutonium and Uranium Nitrate Solution at a Plutonium Fraction of 0.4 in Slab and Cylindrical Geometry

R. C. Lloyd

April 1988

**Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest Laboratory
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CRITICALITY EXPERIMENTS WITH MIXED PLUTONIUM AND URANIUM NITRATE SOLUTION AT A PLUTONIUM FRACTION OF 0.4 IN SLAB AND CYLINDRICAL GEOMETRY

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Fuel Reprocessing Program and the
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Data Development

Pacific Northwest Laboratory
Richland, Washington 99352

SUMMARY

A series of critical experiments was completed with mixed plutonium-uranium solutions having Pu/(Pu + U) ratios of approximately 0.4. 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 105 to 436 g (Pu + U)/liter. The ratio of plutonium to total heavy metal (plutonium plus uranium) was 0.4 for all experiments.

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CRITICALITY EXPERIMENTS WITH MIXED PLUTONIUM AND URANIUM NITRATE SOLUTION AT A PLUTONIUM FRACTION OF 0.4 IN SLAB AND CYLINDRICAL GEOMETRY

1.0 INTRODUCTION

The design and operation of facilities for recycling fast breeder reactor (FBR) 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, and in establishing 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 the 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 $\text{Pu}/(\text{Pu} + \text{U})$ ratios of approximately 0.4, and documents part of the work of the Project's Subtask 120. The experiments were performed in cylindrical and slab geometry. Data were obtained on water reflected, concrete reflected and bare (unreflected) assemblies. The solution concentration was varied from 105 to 436 g $(\text{Pu} + \text{U})/\text{liter}$. These data have application where 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 meters 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 this series of experiments.

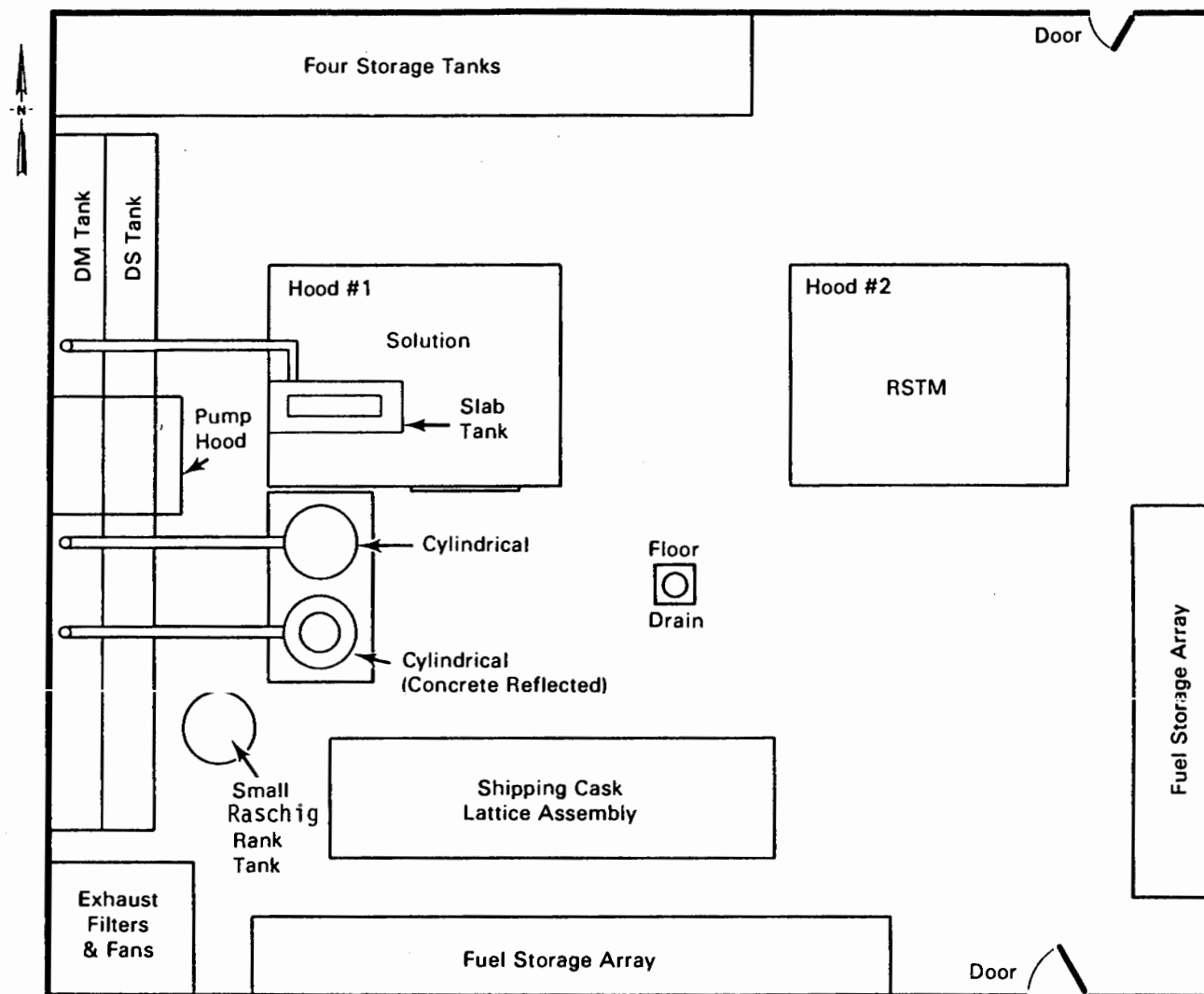


FIGURE 2.1 Floor Schematic Plan of the Critical Assembly Room

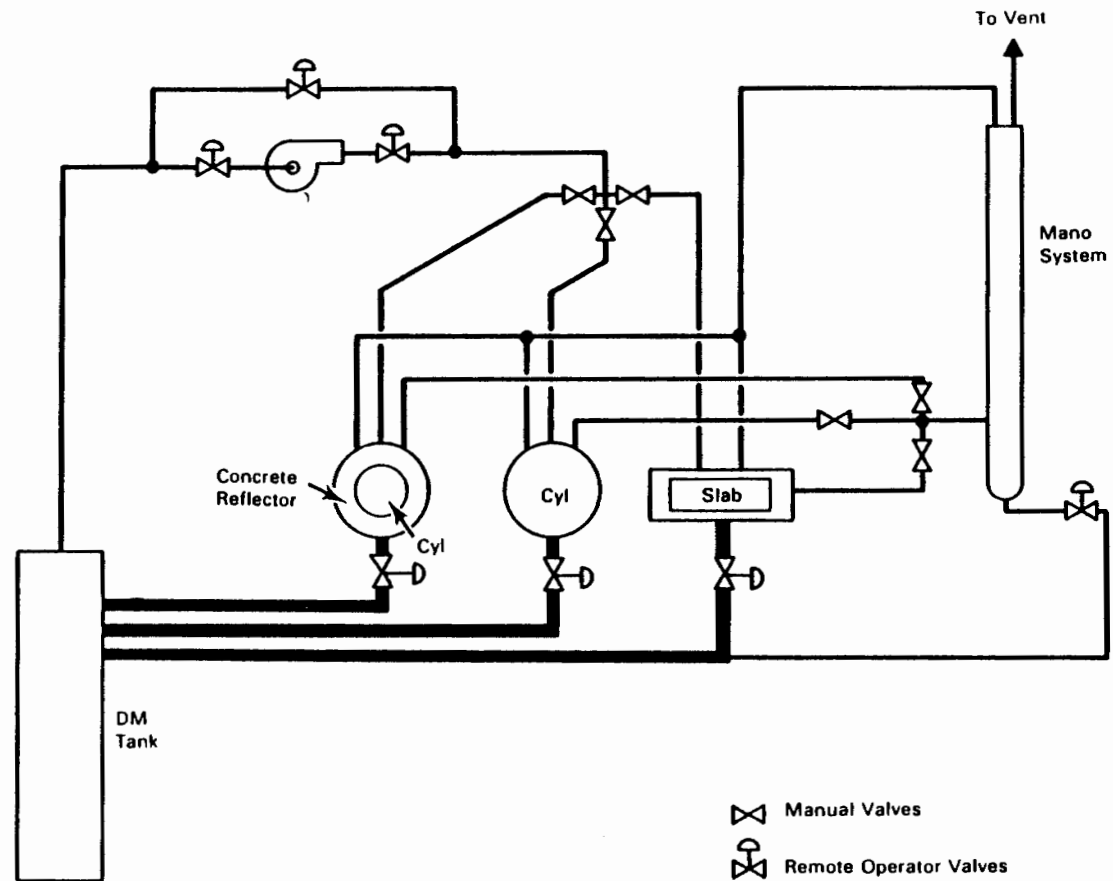


FIGURE 2.2 Piping Schematic for the Three Experimental Vessels

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 cylindrical 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 front 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 for all critical experiments except for CML experiment number 083 in which the large cylinder was replaced by the annular cylinder (53 cm diameter).

The small cylindrical vessel (35.39 cm ID and 106.60 cm inside height) was fabricated of Type 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 reflector tank walls and the 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. The distance between the small cylinder (outside surface) and the bottom of the reflector tank (inside surface) is 16.0 ± 0.2 cm.

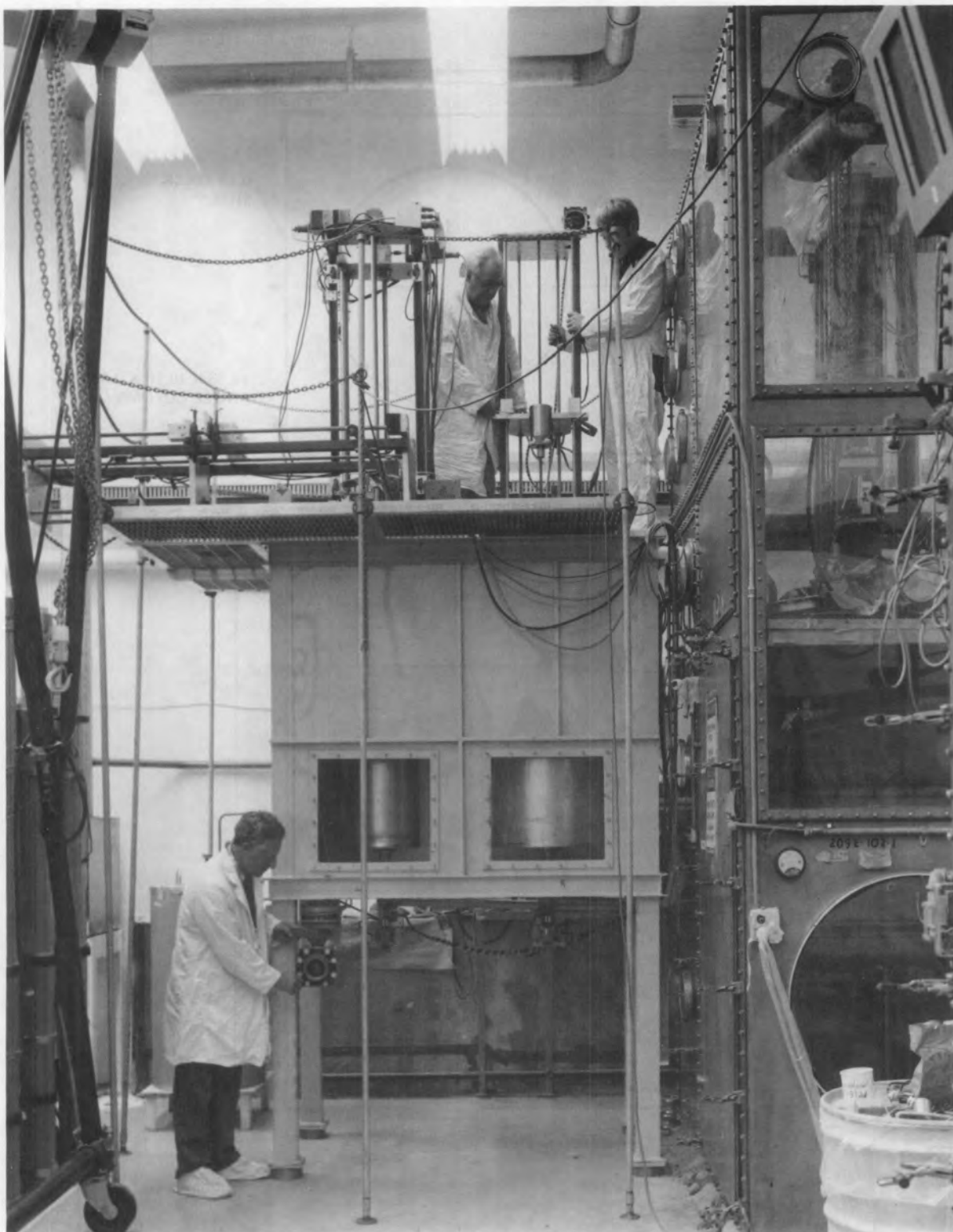


FIGURE 2.3 Photograph of the Cylindrical Vessel System

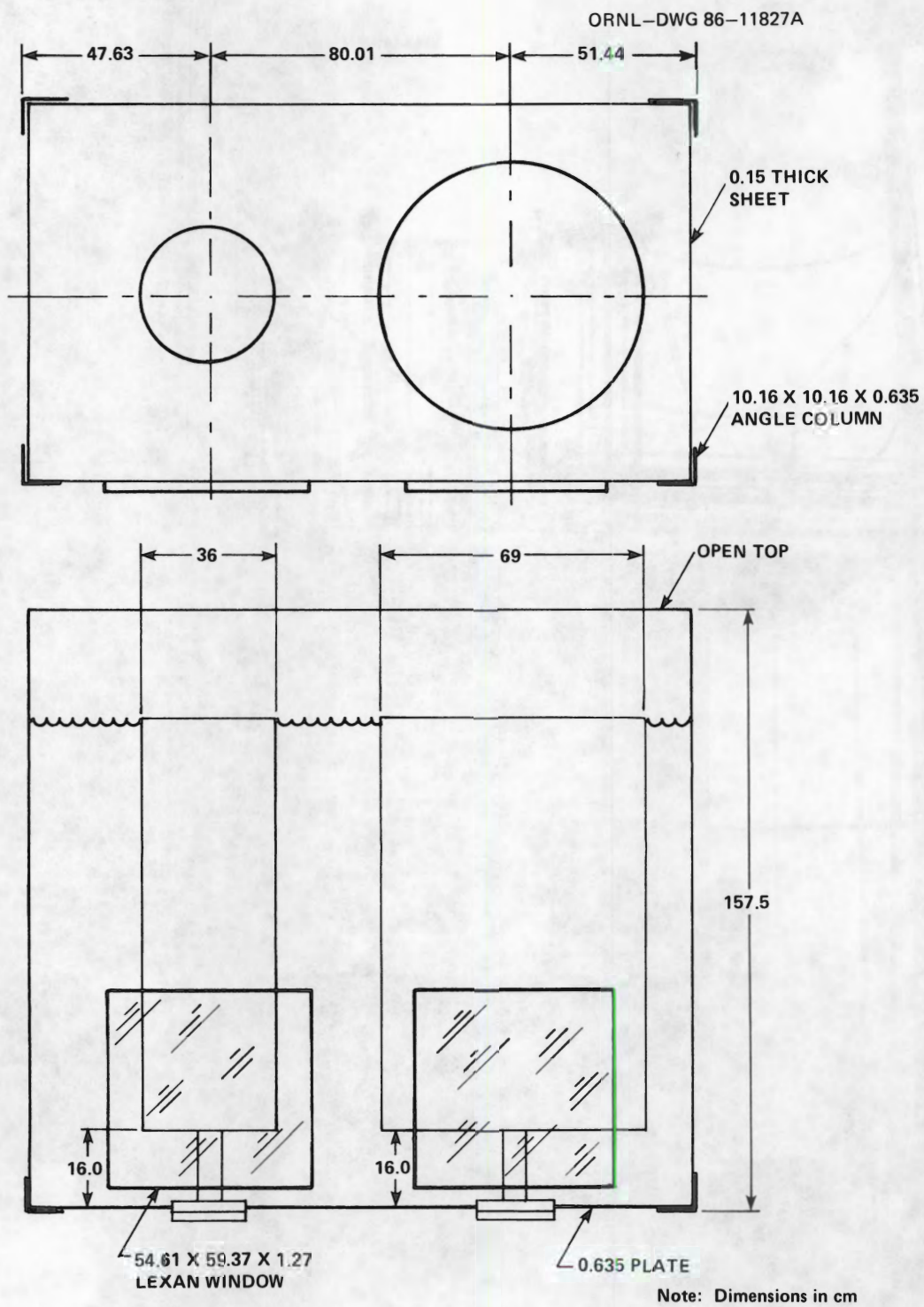


FIGURE 2.4 Placement of Cylindrical Vessels in the Reflector Tank

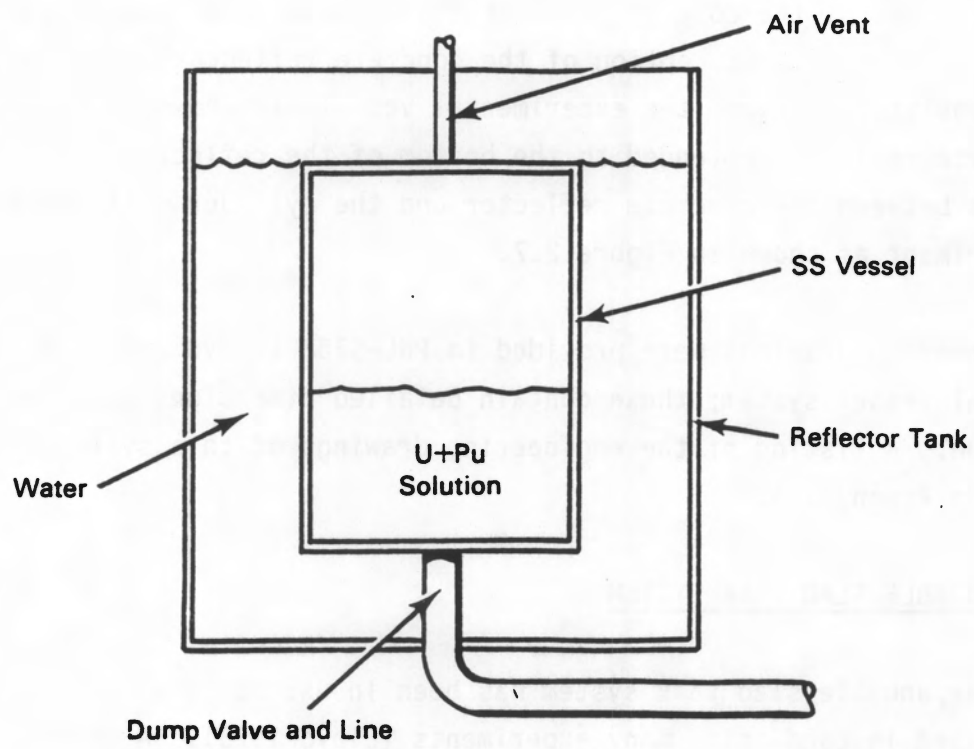


FIGURE 2.5 Schematic of the Cylindrical Vessel

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. The concrete reflector extended to the bottom of the reflector tank. The gap dimensions between the concrete reflector and the cylinder wall varied for each experiment as shown in Figure 2.7.

Engineering drawings were provided in PNL-5768 (Lloyd 1986) for the cylindrical vessel system; these contain detailed dimensions used for fabrication. A listing of the engineering drawings of this system is provided in Appendix A.

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). A listing of the engineering drawings for this system are provided in Appendix B. The engineering drawings were included in PNL-5768, (Lloyd 1986). C. R. Richey has performed an analysis on the grid structure for the slab tank system (Richey 1968) in which he determines its effects on the experiments.

The slab assembly (Figure 2.8) 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.

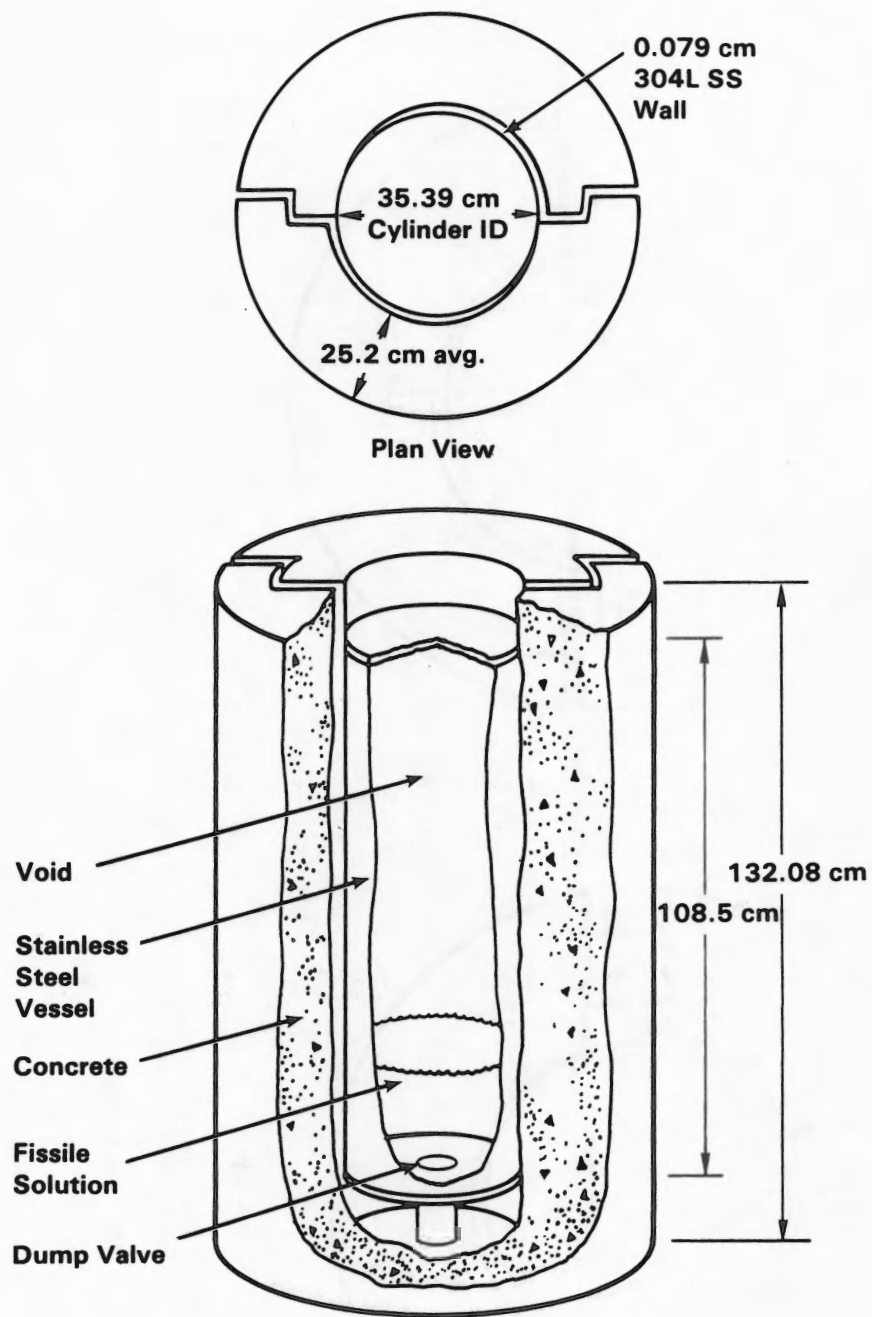
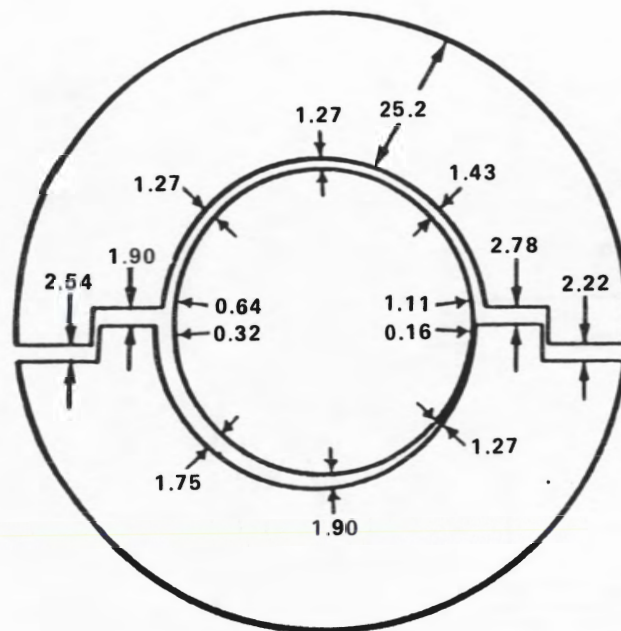


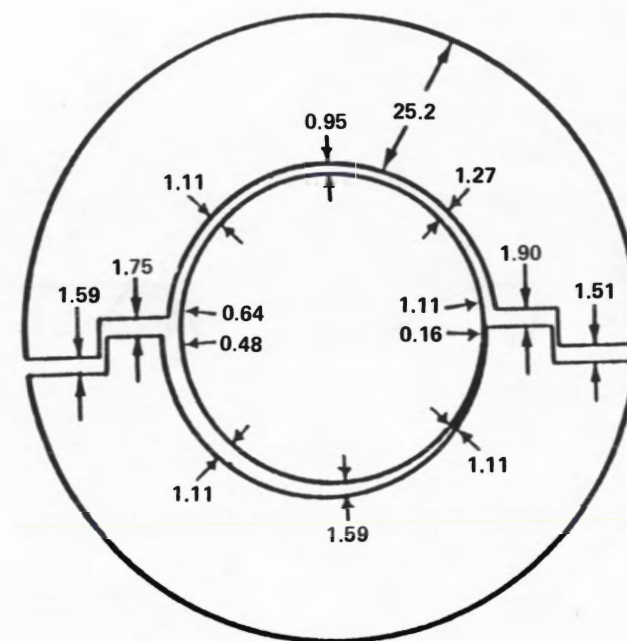
FIGURE 2.6 Schematic of the Concrete Reflected Cylindrical Vessel



PLAN VIEW

Note: Measurements shown were made at vessel top.
Recommended gap at solution level = 0.8 ± 0.2

CML Experiment No. 067,068



PLAN VIEW

Note: Measurements shown were made at vessel top.
Recommended gap at solution level = 0.8 ± 0.2

CML Experiment No. 083

Dimensions in cm

FIGURE 2.7 Gap Dimensions Between Concrete Reflector and Cylinder Wall

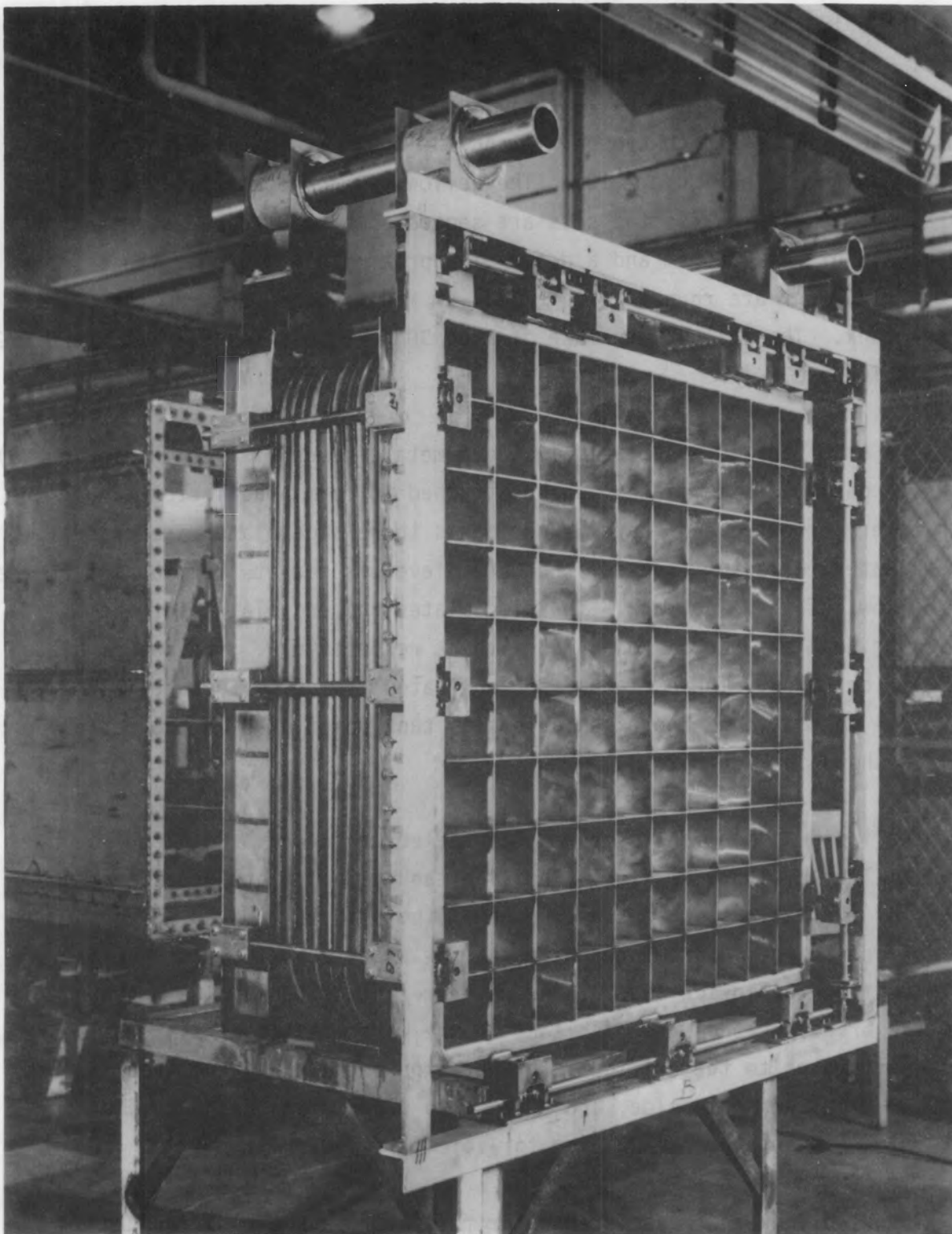


FIGURE 2.8 Photograph of the Slab Assembly

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 Figures 2.8 - 2.10, is constructed with a tip to trough distance of 5.08 cm at a slab thickness of 15.2 cm as shown in Figure 2.10. 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 2.9) 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. 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. The bottom plate is 0.476 cm thick. These plates are fabricated from Type 304L stainless steel. A schematic of the expandable slab tank positioned in the reflector tank is shown in Figure 2.10.

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 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 are 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 2.11). 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.

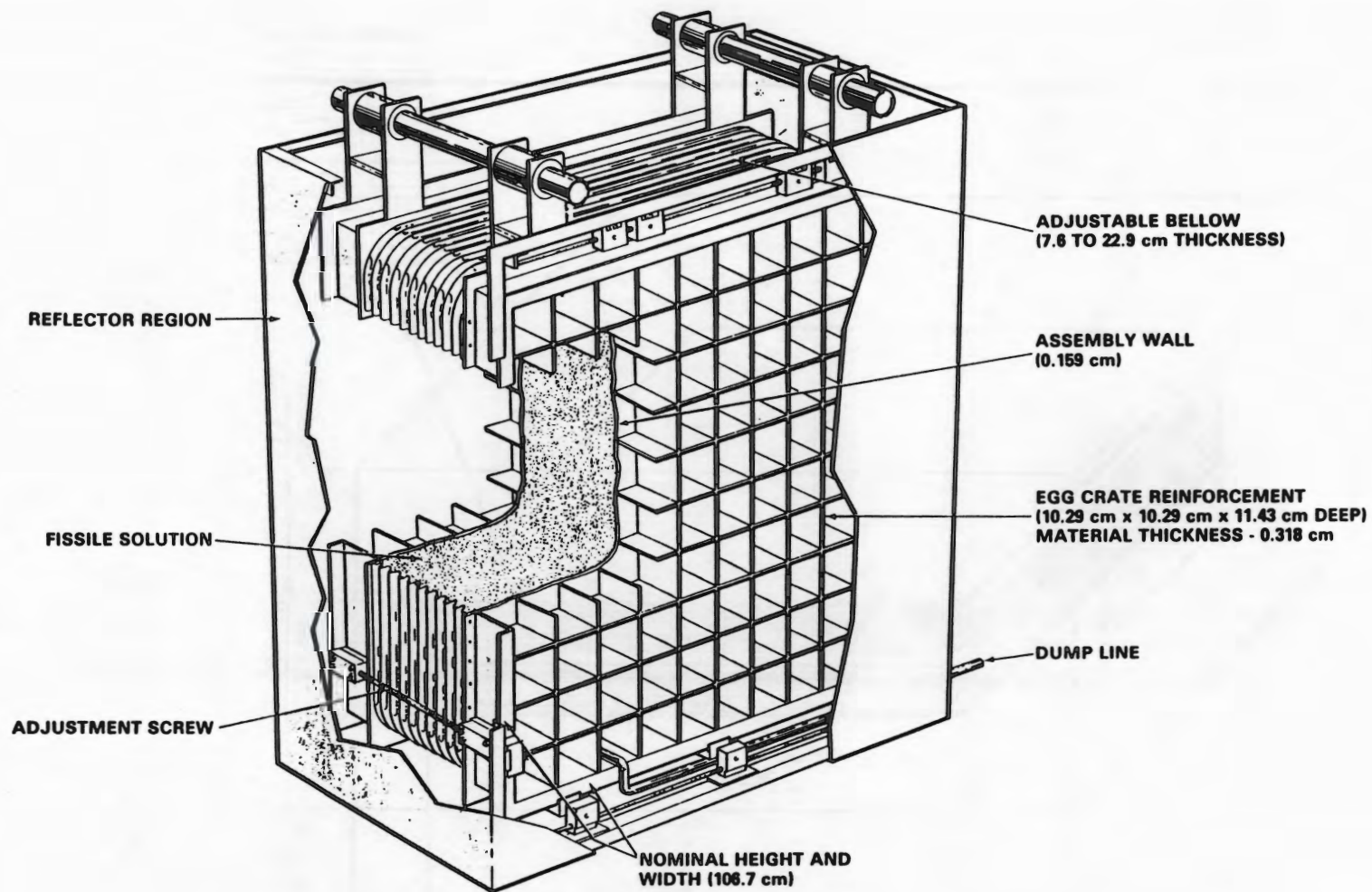


FIGURE 2.9 Critical Experiments Expandable Slab Assembly

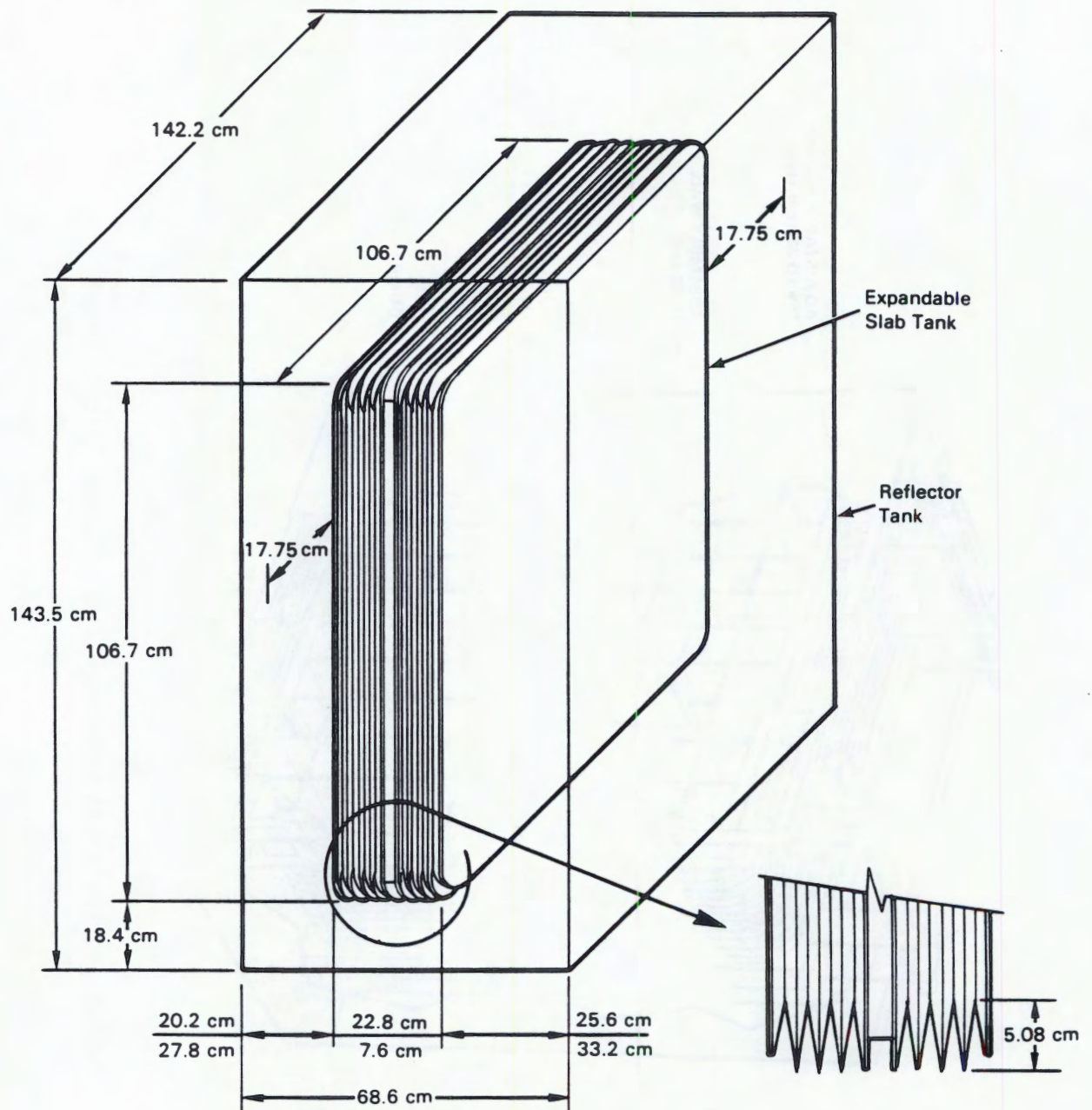


FIGURE 2.10 Schematic of the Expandable Slab Tank Positioned in the Reflector Tank

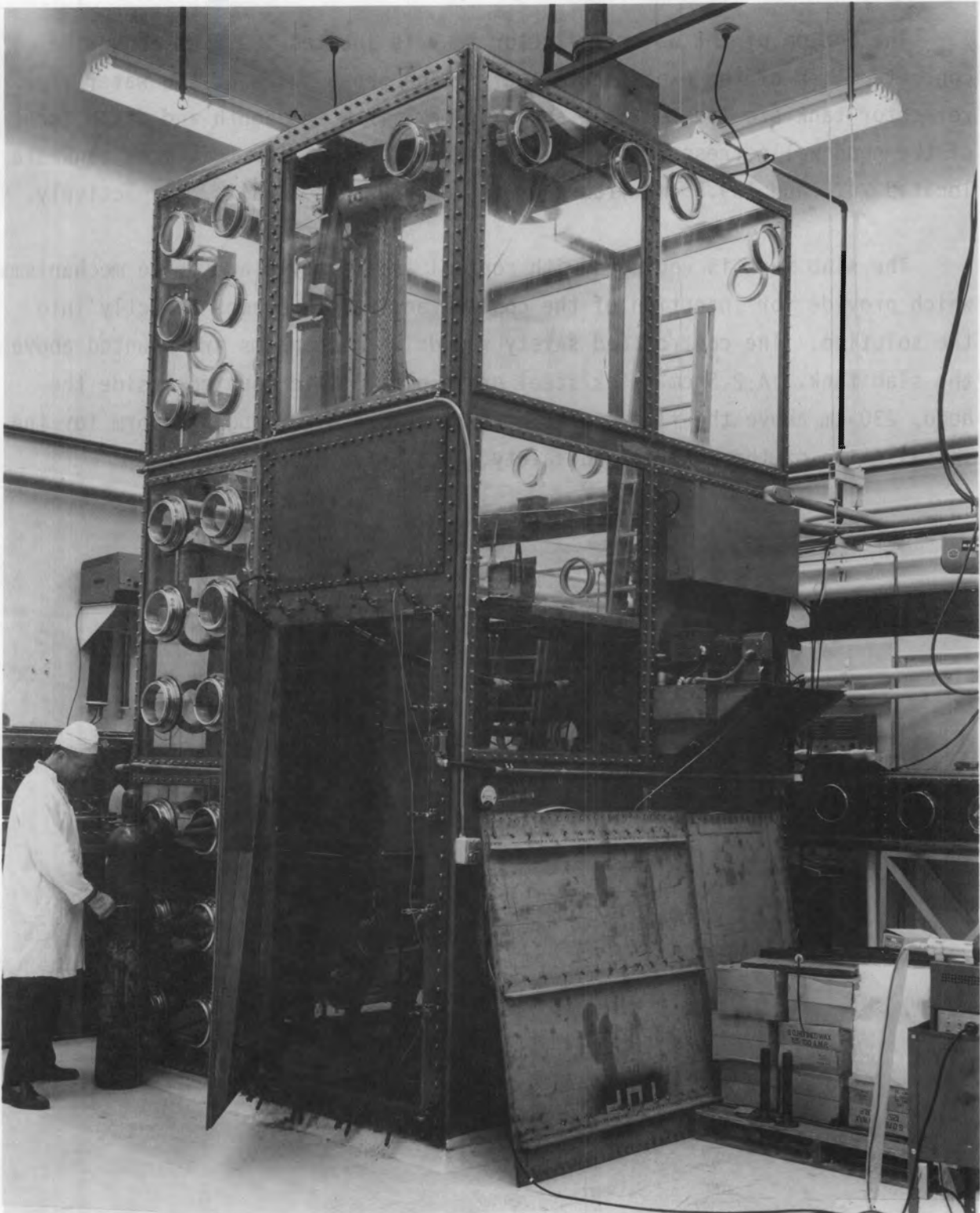


FIGURE 2.11 Containment Hood for Expandable Slab Assembly (Looking at South Side of Slab)

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 safety blade drive mechanisms.

FIGURE 2.11: Containment Hood for Expandable Slab Assembly (Looking at South Side of Slab)

3.0 EXPERIMENTAL RESULTS

This section gives 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, the change in neutron flux is measured 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 greatly increases 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 count rate from the neutron flux is routinely taken on three boron-lined proportional counters located near the experimental vessel. The data from the three counters extrapolate to essentially identical values for solution 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 during the period February - June 1986, when nine experiments were completed with the small cylindrical vessel and eight with the expandable slab experimental system. The data are summarized in Tables 3.1 and 3.2. The indicated critical 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 Company, Hanford Engineering Development Laboratory (HEDL) 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

TABLE 3.1 Criticality Measurements with (Pu + U) Nitrate
Solution in the 35.39 cm Diameter Cylinder

Run Date	Project Case Number	CML Experiment Number	Reflector	Sample Number	Pu (g/liter)	U (g/liter)	Density ^(a) (g/cm ³)	Free Acid (M)	Critical Height (cm)
02/14/86	25	065	Bare	1155	41.69	63.38	1.1749	0.61	44.46
02/19/86	26	066	Water	1156	41.89	63.65	1.1750	0.61	28.11
02/25/86	27	067	Concrete	1161	41.83	63.55	1.1747	0.61	29.36
03/04/86	18	068	Concrete	1163	118.71	173.98	1.4638	1.02	27.03
03/07/86	19	069	Water	1164	119.04	174.67	1.4645	1.05	25.26
03/11/86	20	070	Bare	1165	118.90	174.53	1.4645	1.03	41.08
04/25/86	15	077	Bare	1174	172.56	262.79	1.6706	1.23	57.97
04/30/86	16	078	Water	1177	172.82	262.55	1.6705	1.23	28.93
06/05/86	17	083	Concrete	1184	173.22	262.88	1.6704	1.22	30.60

(a) Density measured at 23°C

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

Run Date	Project Case Number	CML Experiment Number	Reflector	Sample Number	Pu (g/liter)	U (g/liter)	Density ^(a) (g/cm ³)	Free Acid (M)	Slab Thickness (cm)	Critical Height (cm)
02/04/86	23	063	Bare (Both Sides Reflector Tank)	1153	41.06	62.89	1.1725	0.61	19.81	54.70
02/10/86	24	064	Water	1154	41.90	63.36	1.1752	0.61	13.97	80.14
03/13/86	21	071	Water	1166	118.87	173.48	1.4633	1.03	13.97	47.44
03/14/86	21	072	Water	1166	118.87	173.48	1.4633	1.03	12.70	67.16
03/17/86	22	073	Bare (Both Sides Reflector Tank)	1167	119.04	174.01	1.4637	1.04	17.02	(b)
03/18/86	22	074	Bare (Both Sides Reflector Tank)	1167	119.04	174.01	1.4637	1.04	18.03	70.13
04/22/86	13	075	Bare (Both Sides Reflector Tank)	1170	172.77	262.81	1.6703	1.24	19.05	85.38
04/23/86	14	076	Water	1171	172.68	262.94	1.6699	1.24	13.46	74.19

(a) Density measured at 23°C

(b) Data show criticality not possible in the slab with 17.02 cm thickness with Pu + U nitrate being used in this experiment.

TABLE 3.3 Chemical Analyses Methods

<u>Measurement</u>	<u>Method Title^(a)</u>	<u>Date of 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 Uranium/Plutonium Solutions. (Using an improved oxalate method) (40.22)	02/04/86
Density	Density of Solutions. (Using Mettler/ Paar Density Meter) (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
Impurities	ICP Analysis (SP-7B)	04/01/86

(a) The numbers in brackets are HEDL's method numbers.

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).

TABLE 3.4 Chemical Analysis Values for Americium-241

Sample Number	²⁴¹ Am (ug/ml)	Analysis Date
1153	207	02/12/86
1161	211	05/29/86
1166	601	03/28/86
1170	932	05/02/86
1174	897	05/02/86
1177	896	05/12/86
1184	897	06/20/86

Sample 1153 and 1161 covers experiments 063 - 067.

Sample 1166 covers experiments 068 - 074.

Samples 1170, 1174 and 1177 covers experiments 075 - 078.

Sample 1184 covers experiment 083.

TABLE 3.5 Isotopic Analyses Values of Pu and U^(a)

	Sample 1153 ^(b)	Sample 1161 ^(b)	Sample 1166 ^(c)	Sample 1170 ^(d)	Sample 1174 ^(d)	Sample 1177 ^(d)	Sample 1184 ^(e)
Pu ^(f)	(2-13-86)	(3-11-86)	(3-31-86)	(5-5-86)	(5-5-86)	(5-14-86)	(6-23-86)
238	0.029 ± 0.003	0.029 ± 0.003	0.027 ± 0.002	0.030 ± 0.001	0.029 ± 0.001	0.029 ± 0.001	0.028 ± 0.001
239	91.13 ± 0.04	91.11 ± 0.04	91.10 ± 0.04	91.14 ± 0.04	91.15 ± 0.04	91.11 ± 0.04	91.12 ± 0.04
240	8.29 ± 0.04	8.31 ± 0.04	8.29 ± 0.04	8.33 ± 0.04	8.28 ± 0.04	8.32 ± 0.04	8.32 ± 0.04
241	0.455 ± 0.005	0.457 ± 0.004	0.451 ± 0.003	0.453 ± 0.004	0.449 ± 0.004	0.453 ± 0.004	0.445 ± 0.003
242	0.094 ± 0.002	0.096 ± 0.003	0.090 ± 0.002	0.093 ± 0.002	0.095 ± 0.002	0.093 ± 0.002	0.093 ± 0.002
U ^(g)	(2-19-86)	(3-11-86)	(3-31-86)	(5-5-86)	(5-5-86)	(5-13-86)	(6-24-86)
238	99.414 ± 0.008	99.404 ± 0.004	99.403 ± 0.005	99.403 ± 0.003	99.404 ± 0.003	99.402 ± 0.005	99.408 ± 0.004
236	0.023 ± 0.002	0.024 ± 0.001	0.022 ± 0.002	0.024 ± 0.001	0.024 ± 0.001	0.024 ± 0.002	0.023 ± 0.002
235	0.555 ± 0.006	0.564 ± 0.004	0.569 ± 0.004	0.565 ± 0.003	0.565 ± 0.003	0.568 ± 0.004	0.564 ± 0.003
234	0.007 ± 0.002	0.007 ± 0.001	0.006 ± 0.001	0.008 ± 0.001	0.007 ± 0.001	0.007 ± 0.002	0.006 ± 0.001

(a) All values given in wt%

(b) Samples 1153 and 1161 are for experiments 063 - 067

(c) Sample 1166 is for experiments 068 - 074

(d) Samples 1170, 1174 and 1177 are for experiments 075 - 078

(e) Sample 1184 is for experiment 083

(f) Date of Pu analysis

(g) Date of U analysis.

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

	Experiment Number	Temperature °C			Reflector Level Distance Below Vessel Top (cm)	Control and Safety Blade Distance Below Vessel Top (cm)
		Room	Storage Tank	Reflector		
<u>Cylinder</u>	065	22.7	18.9	NA	NA	(b)
	066	22.5	18.8	13.6	2.54	1.27
	067	23.7	18.8	23.0	(a)	1.27
	068	24.1	20.8	23.5	(a)	1.27
	069	24.6	21.6	18.6	2.54	1.27
	070	24.6	22.3	NA	NA	(b)
	077	21.3	24.4	NA	NA	(b)
	078	22.3	25.1	19.6	2.54	1.27
	083	23.2	27.2	22.7	(a)	1.27
		Hood				
<u>Slab</u>	063	23.1	19.1	NA	NA	5
	064	22.5	18.8	17.2	2.54	5
	071	24.7	24.6	22.1	2.54	5
	072	24.9	24.5	22.7	2.54	5
	073	24.9	24.3	NA	NA	5
	074	25.1	24.6	NA	NA	5
	075	22.1	24.1	NA	NA	5
	076	22.2	24.6	21.7	2.54	5

NA - Not applicable

(a) Top of concrete reflector was 7.6 cm above the top of the experimental vessel and extended to the floor of the reflector tank.

(b) Control and safety blade top was 1.9 cm below tank bottom.

3.3 SOURCES OF ERROR

It is very difficult 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. An evaluation of this type was done for the variables involved in earlier experiments and has been reported (Primm 1986). From Primm's analysis it was found that the principal uncertainty, or error, was caused by the uncertainty in the free acid values. Since then, a study was made of chemical analysis methods and a free acid analysis method developed and reported (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 included the critical height, plutonium concentration, uranium concentration, density, free acid and composition of reflectors. It was recognized by Primm 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 Estimate of Measurement Uncertainties

Pu Concentration	± 0.2%
U Concentration	± 0.2%
Density	± 0.0003 g/cm ³
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 from three separate curves would indicate a smaller value as reasonable. The 1.6 mm is the smallest unit on the sight tube.

4.0 ACKNOWLEDGMENTS

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.

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

5.0 REFERENCES

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APPENDIX A

A LISTING OF ENGINEERING DRAWINGS FOR THE CYLINDRICAL VESSEL SYSTEM

APPENDIX A

A LISTING OF ENGINEERING DRAWINGS FOR THE CYLINDRICAL VESSEL SYSTEM

CFRP Assembly H-2-33856, Sheet 1 of 5

CFRP H₂O Tank and Cover H-2-33856, Sheet 2 of 5

CFRP Process Tanks H-2-33856, Sheet 3 of 5

CFRP Tank Covers and Shield H-2-33856, Sheet 4 of 5

CFRP Dump Valve H-2-33856, Sheet 5 of 5

APPENDIX B

A LISTING OF ENGINEERING DRAWINGS FOR THE EXPANDABLE SLAB TANK SYSTEM

APPENDIX B

A LISTING OF ENGINEERING DRAWINGS FOR THE EXPANDABLE SLAB TANK SYSTEM

Tank Assembly H-2-32570, Sheet 1 of 8
Bellows Tank Side Plates H-2-32570, Sheet 2 of 8
Bellows Tank Reinforcing H-2-32570, Sheet 3 of 8
Tank Detail H-2-32570, Sheet 4 of 8
Bellows Tank Detail H-2-32570, Sheet 5 of 8
Bellows Tank Detail H-2-32570, Sheet 6 of 8
Bellows Tank Hood Arrangement H-2-32570, Sheet 7 of 8
Bellows Tank Dump Line Assembly H-2-32570, Sheet 8 of 8

APPENDIX C

LEAST SQUARE FITS OF THE CRITICAL APPROACH DATA

APPENDIX C

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 being located at the top of the bellows. The solution heights for all least square plots are in inches.

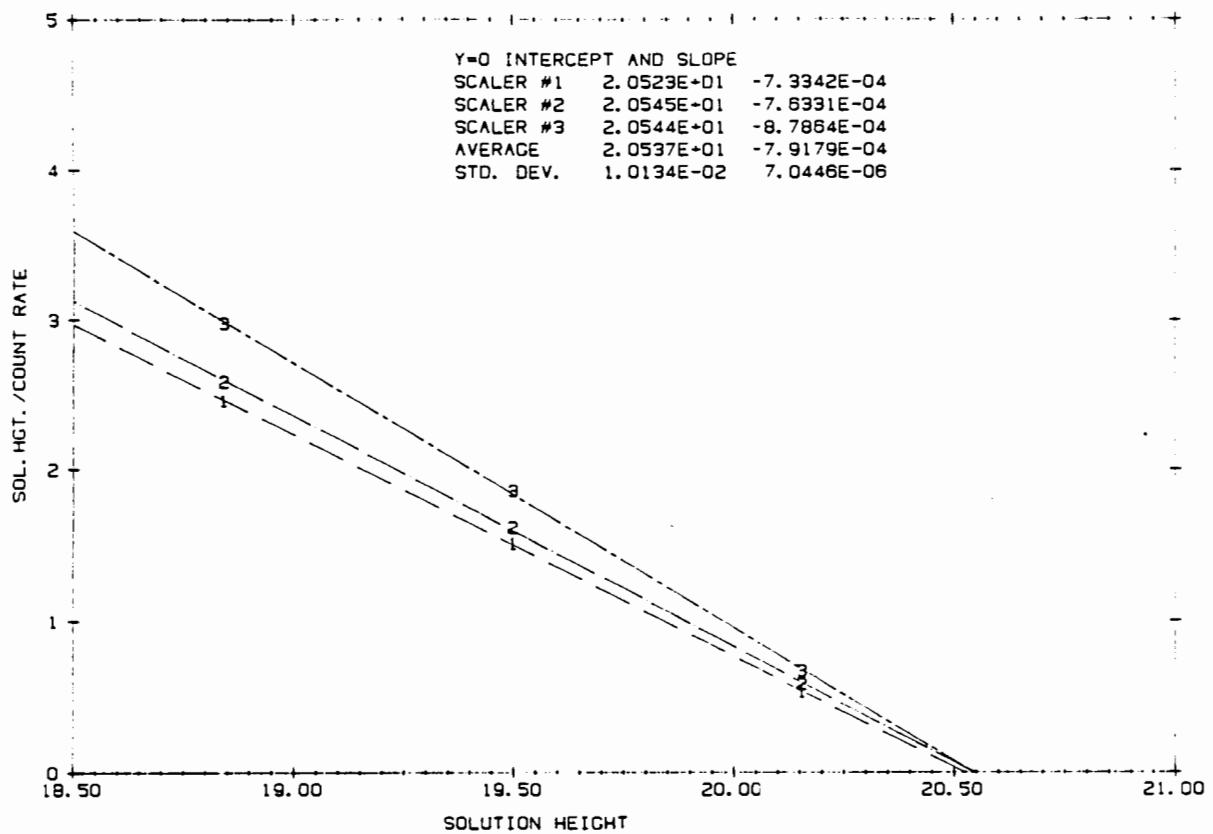


FIGURE C.1 Least Square Fits for CFRP-PNC 063

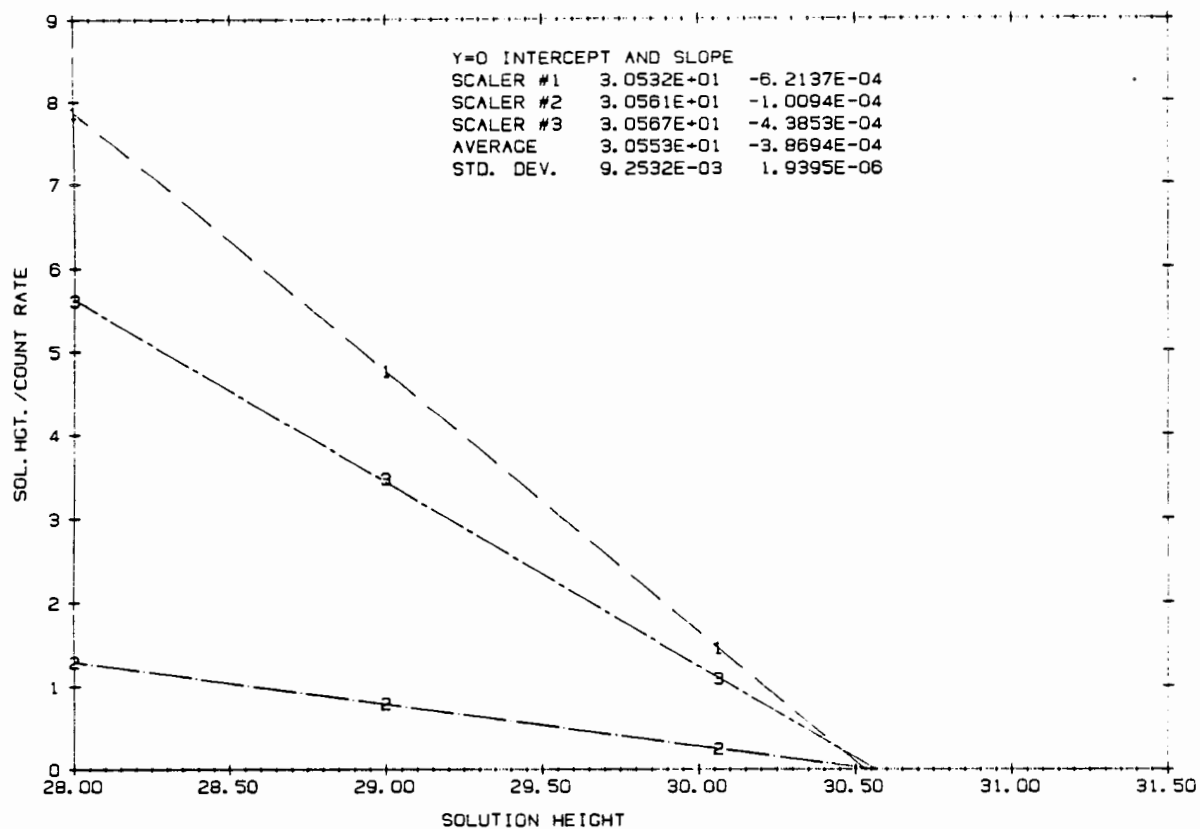


FIGURE C.2 Least Square Fits for CFRP-PNC 064

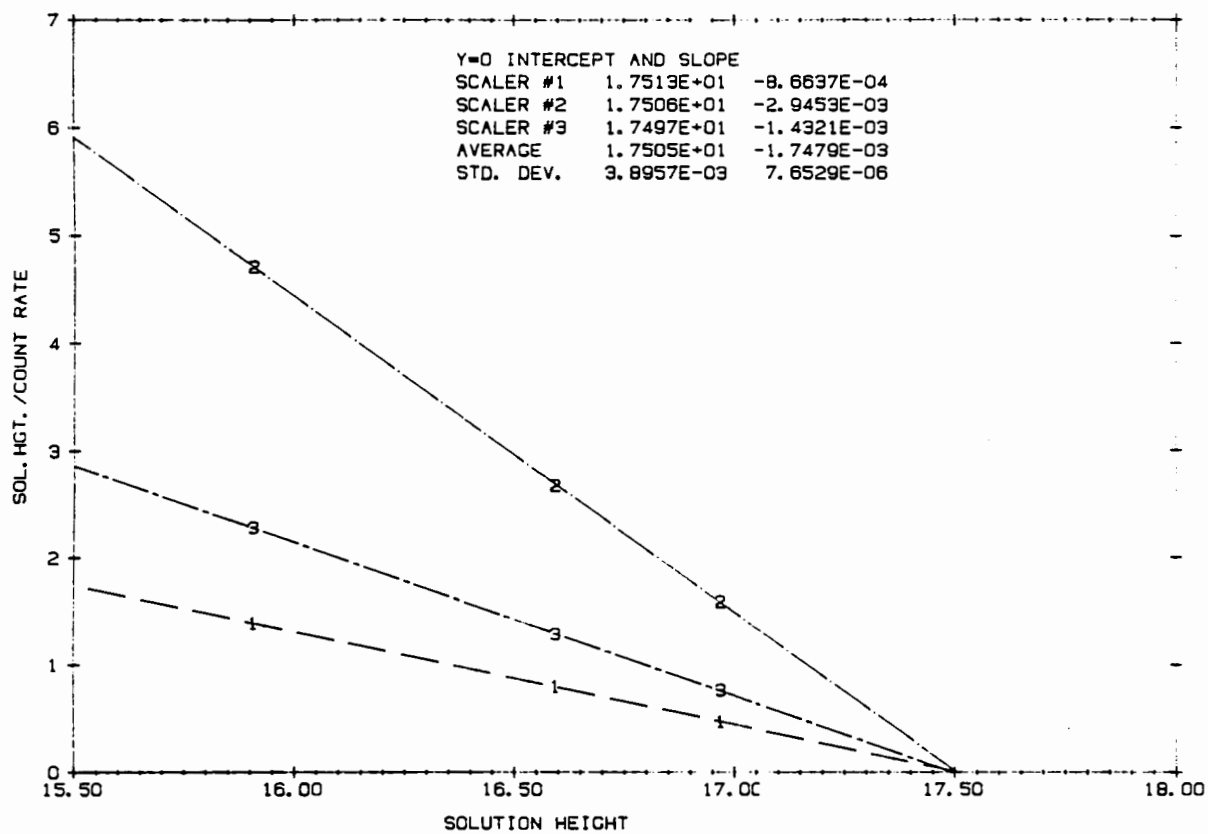


FIGURE C.3 Least Square Fits for CFRP-PNC 065

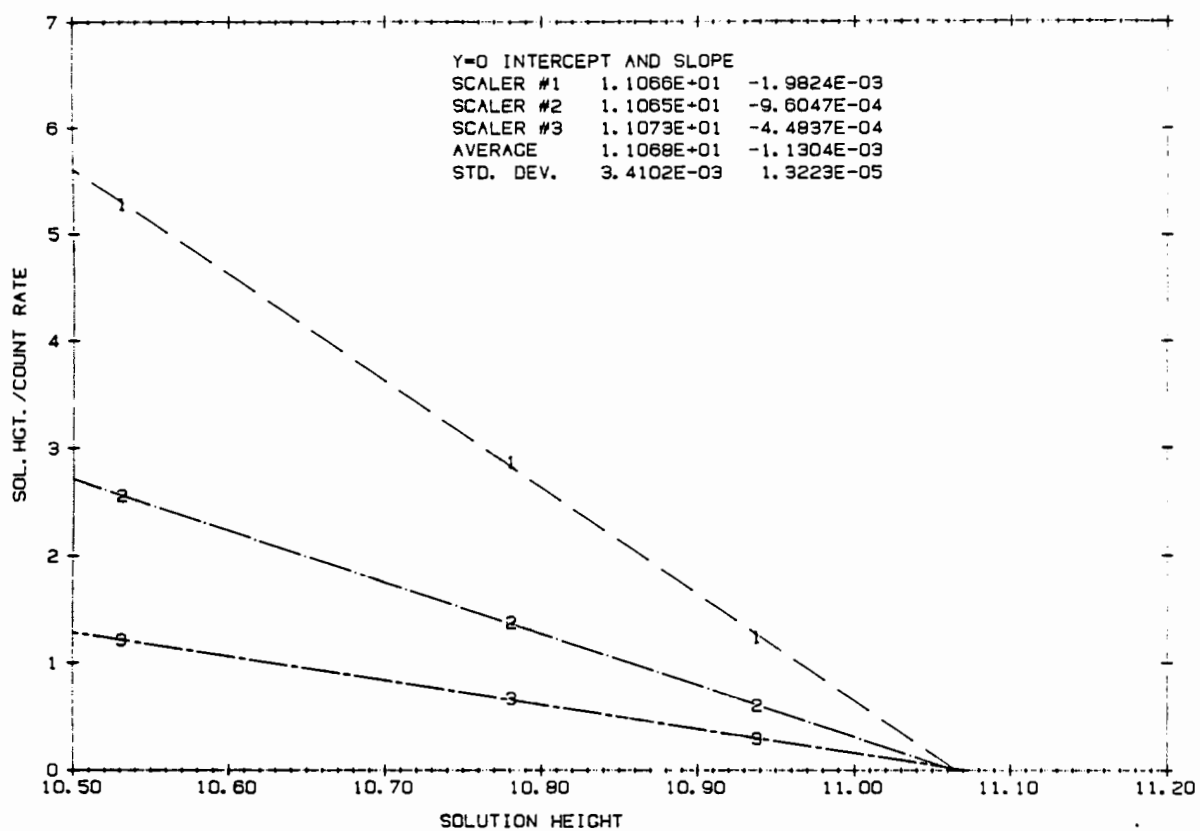


FIGURE C.4 Least Square Fits for CFRP-PNC 066

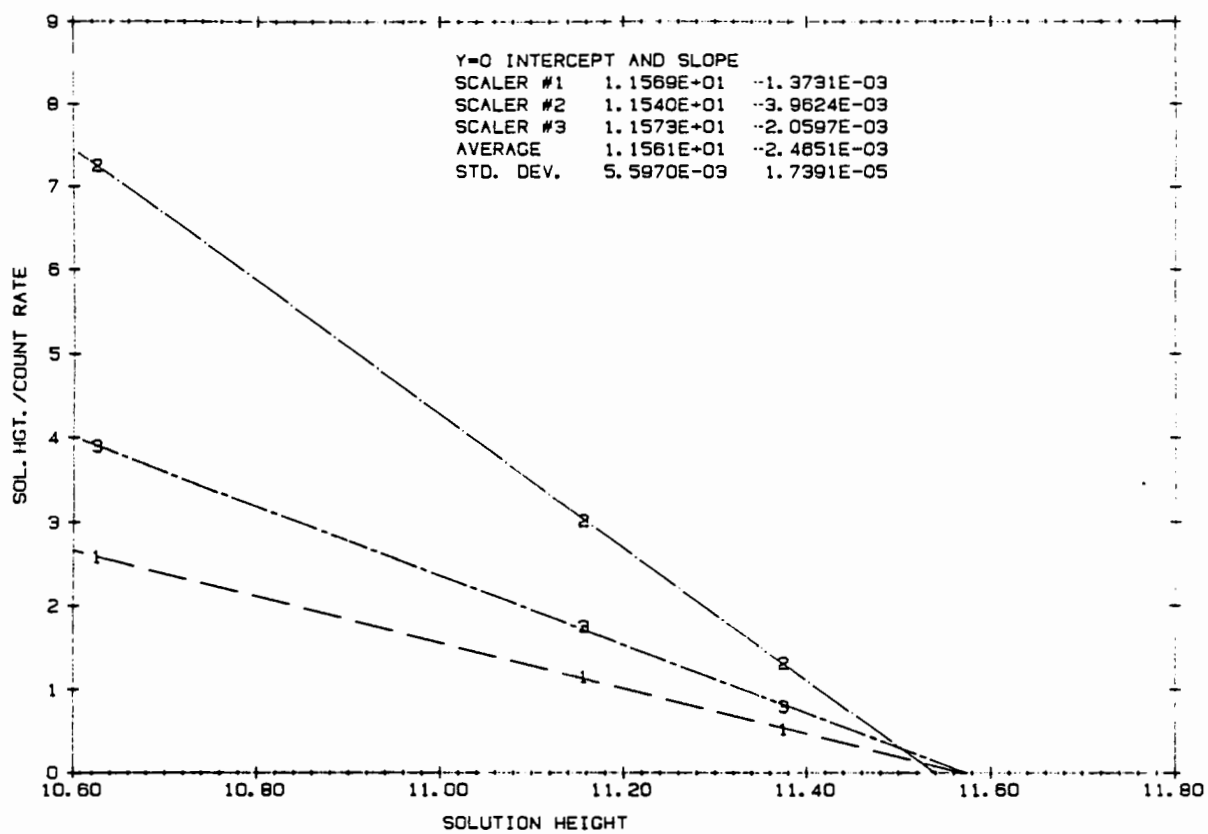


FIGURE C.5 Least Square Fits for CFRP-PNC 067

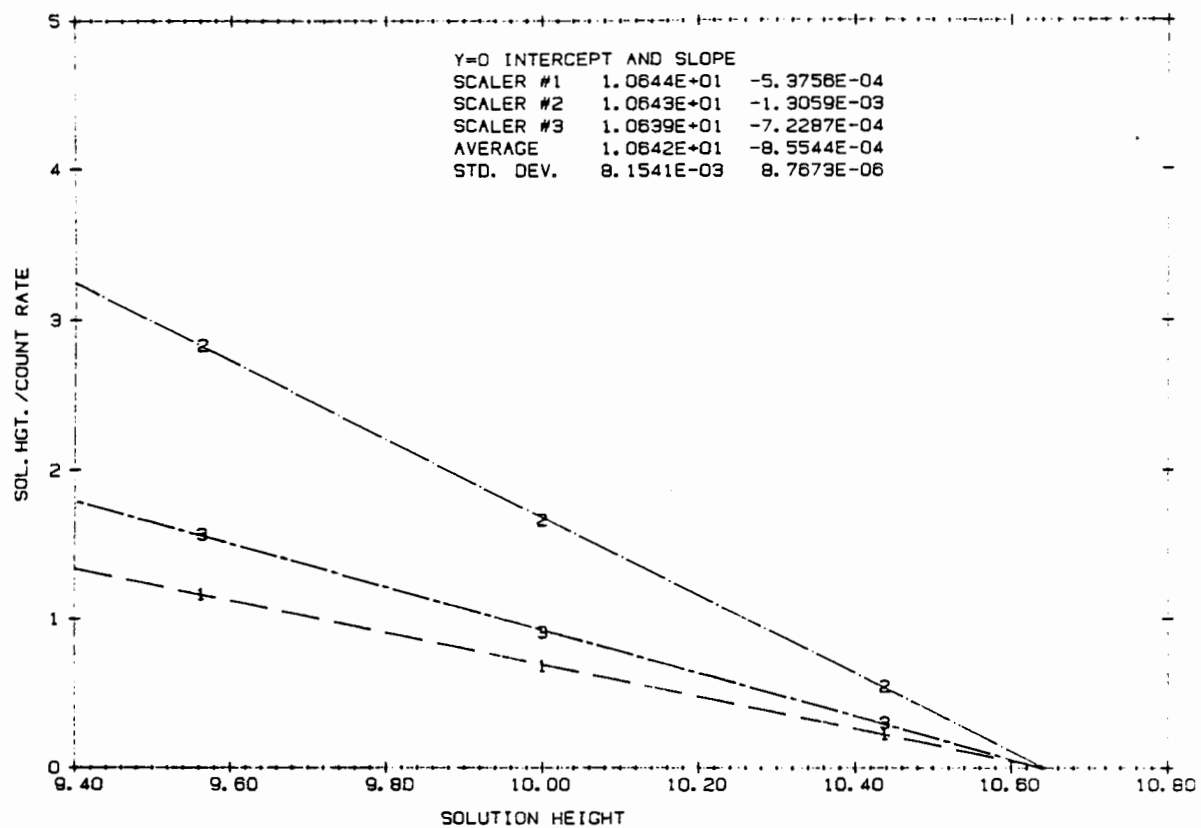


FIGURE C.6 Least Square Fits for CFRP-PNC 068

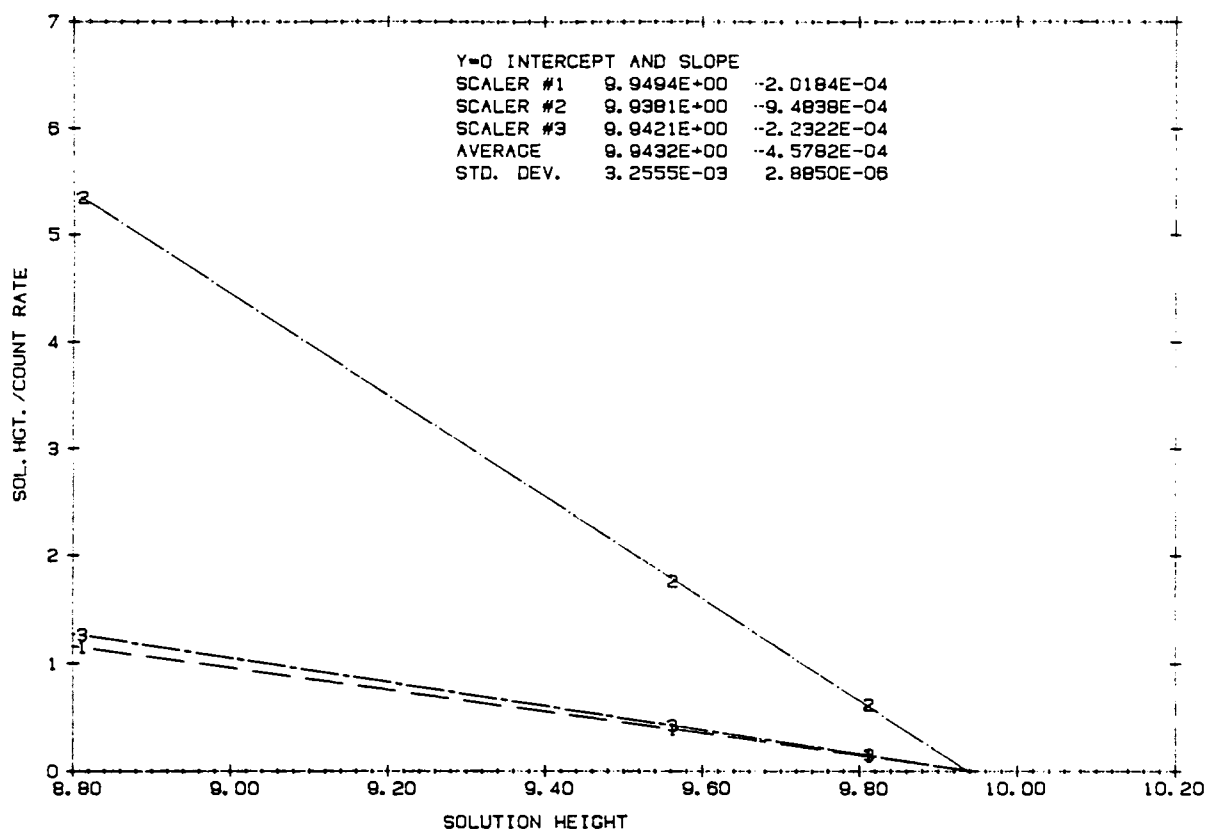


FIGURE C.7 Least Square Fits for CFRP-PNC 069

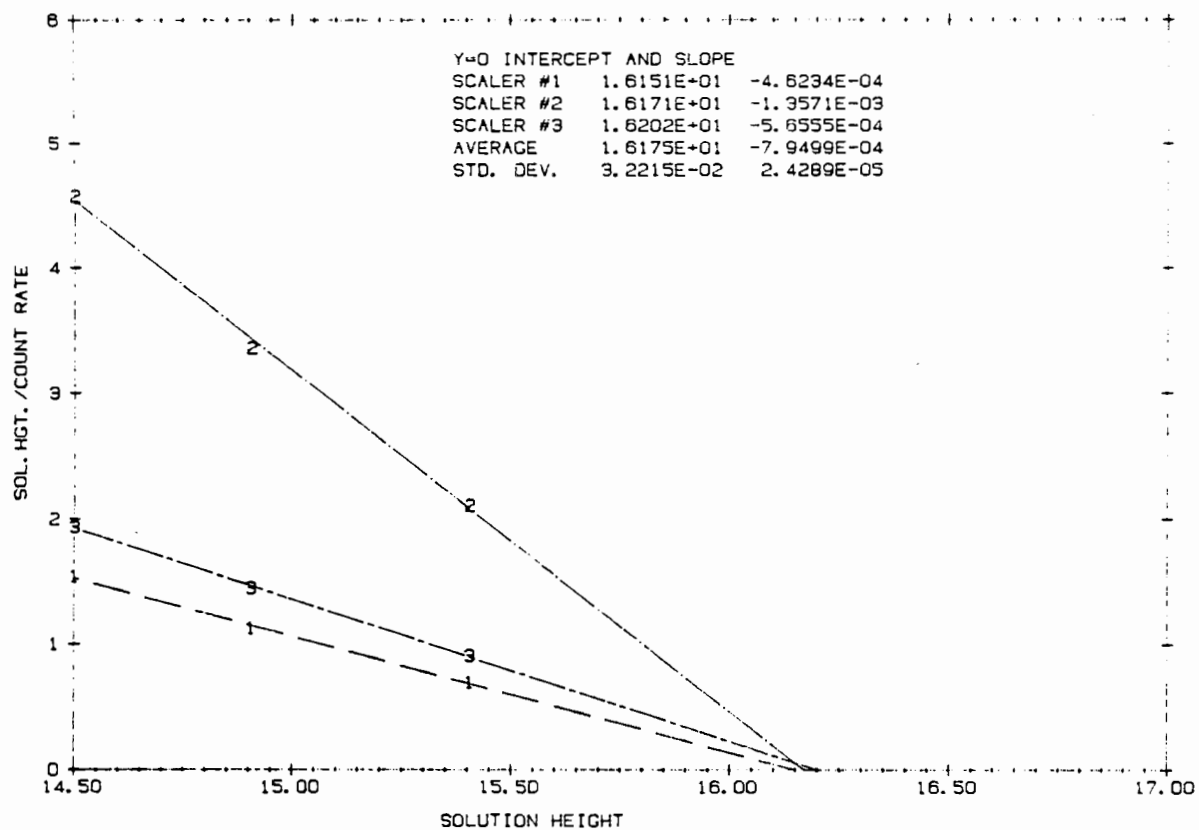


FIGURE C.8 Least Square Fits for CFRP-PNC 070

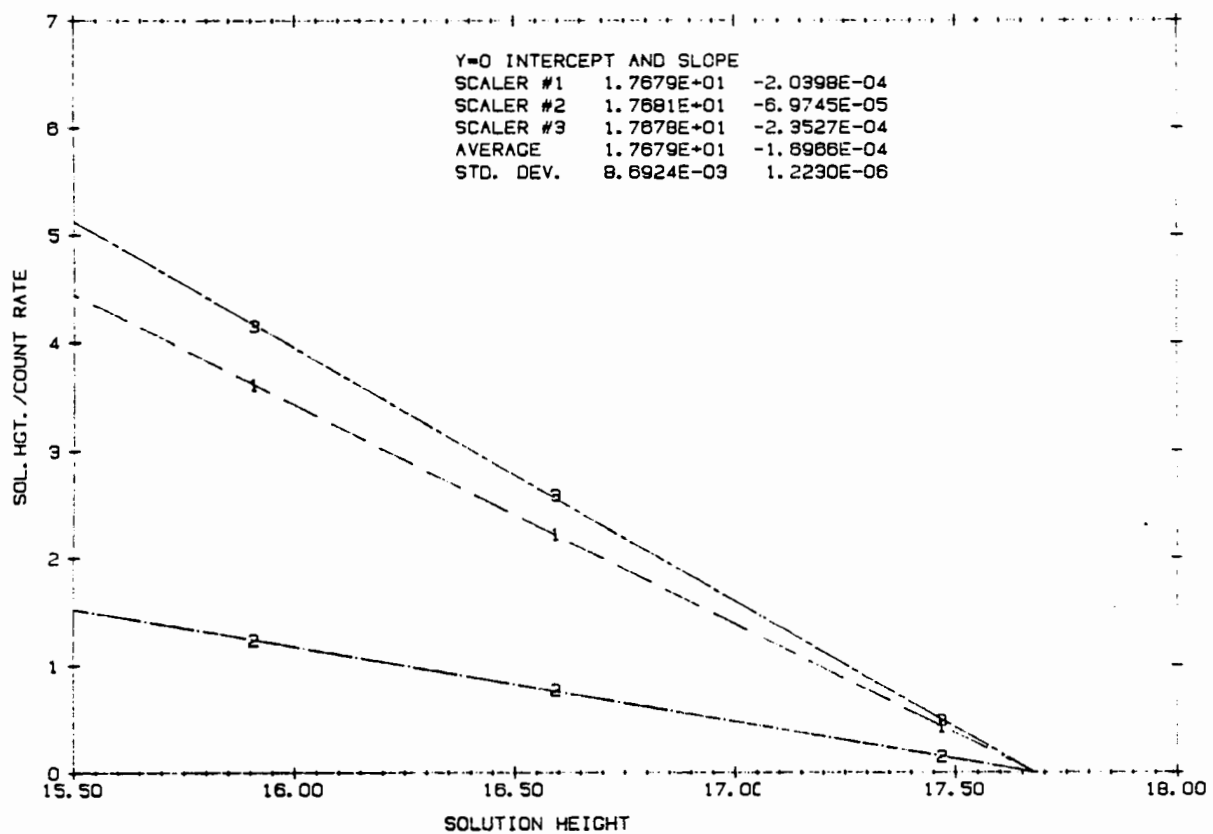


FIGURE C.9 Least Square Fits for CFRP-PNC 071

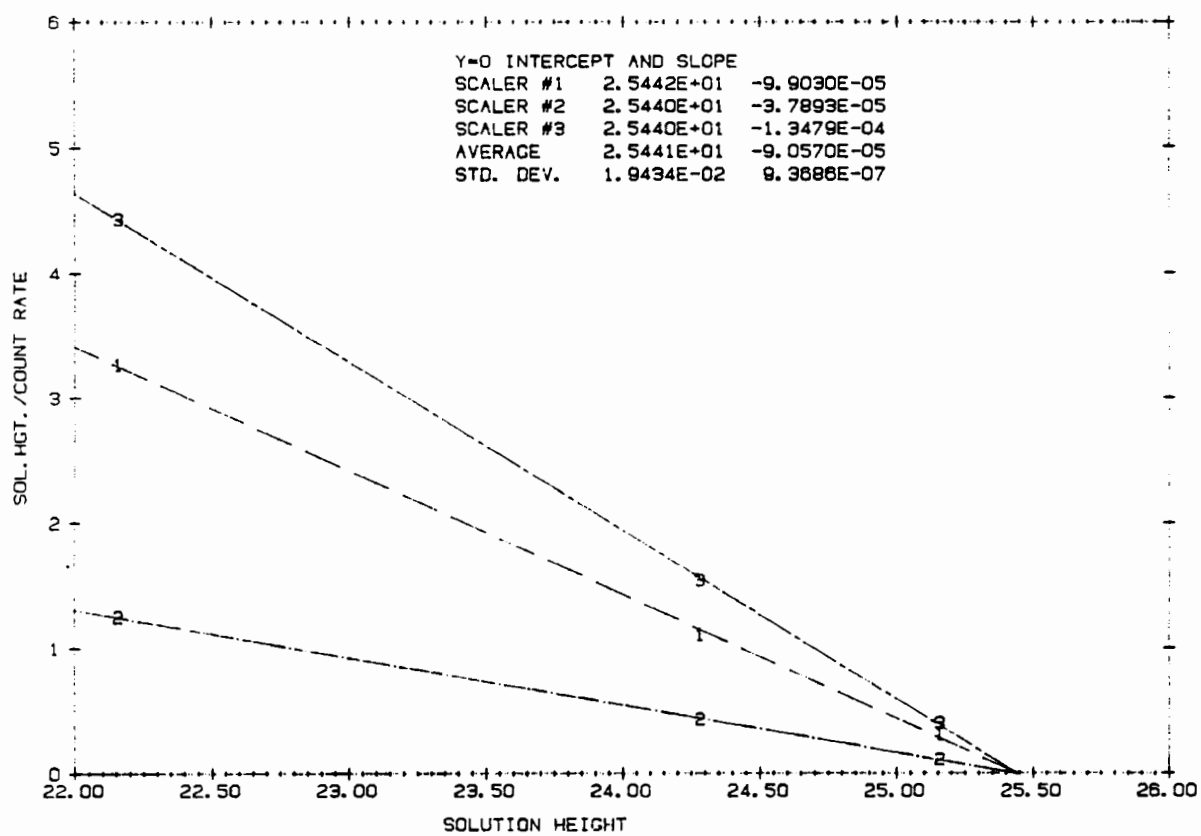


FIGURE C.10 Least Square Fits for CFRP-PNC 072

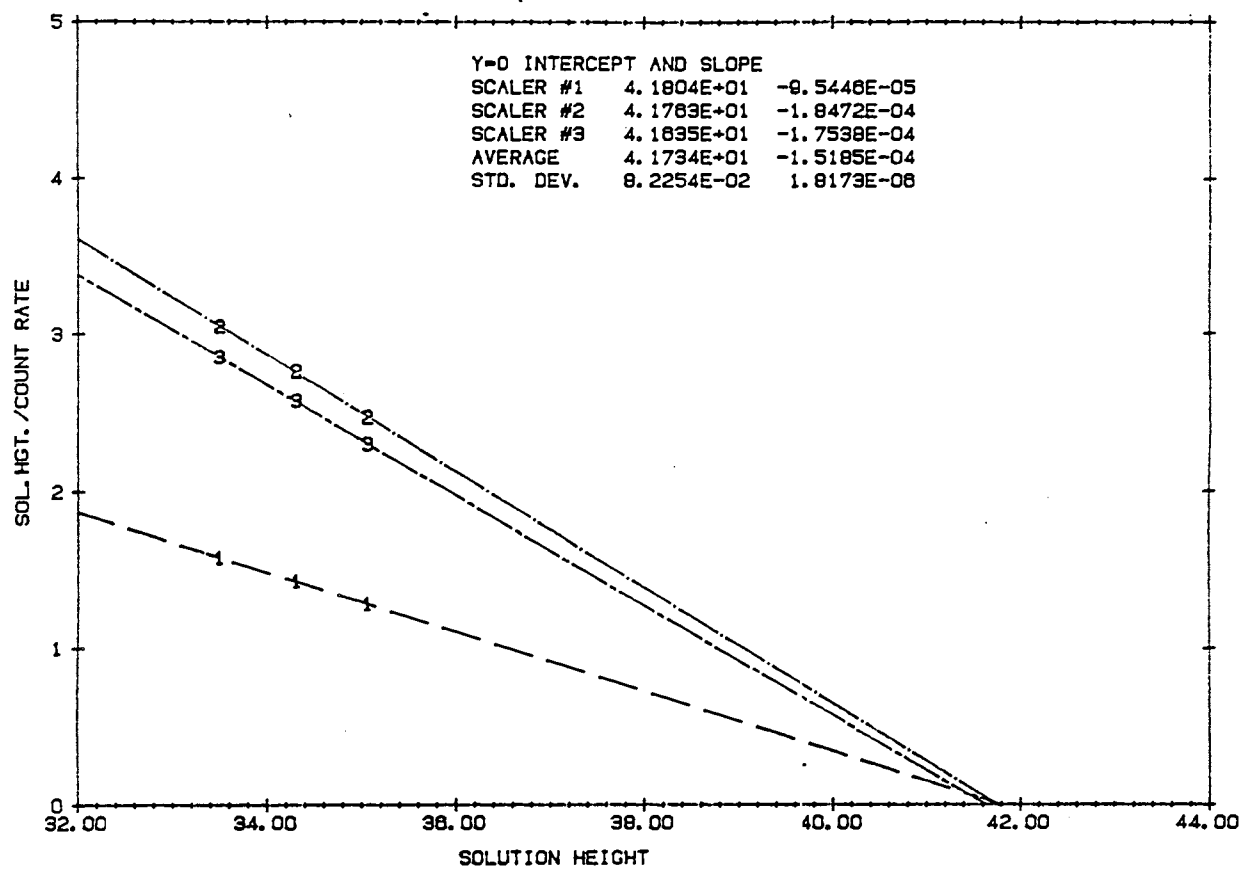


FIGURE C.11 Least Square Fits for CFRP-PNC 073

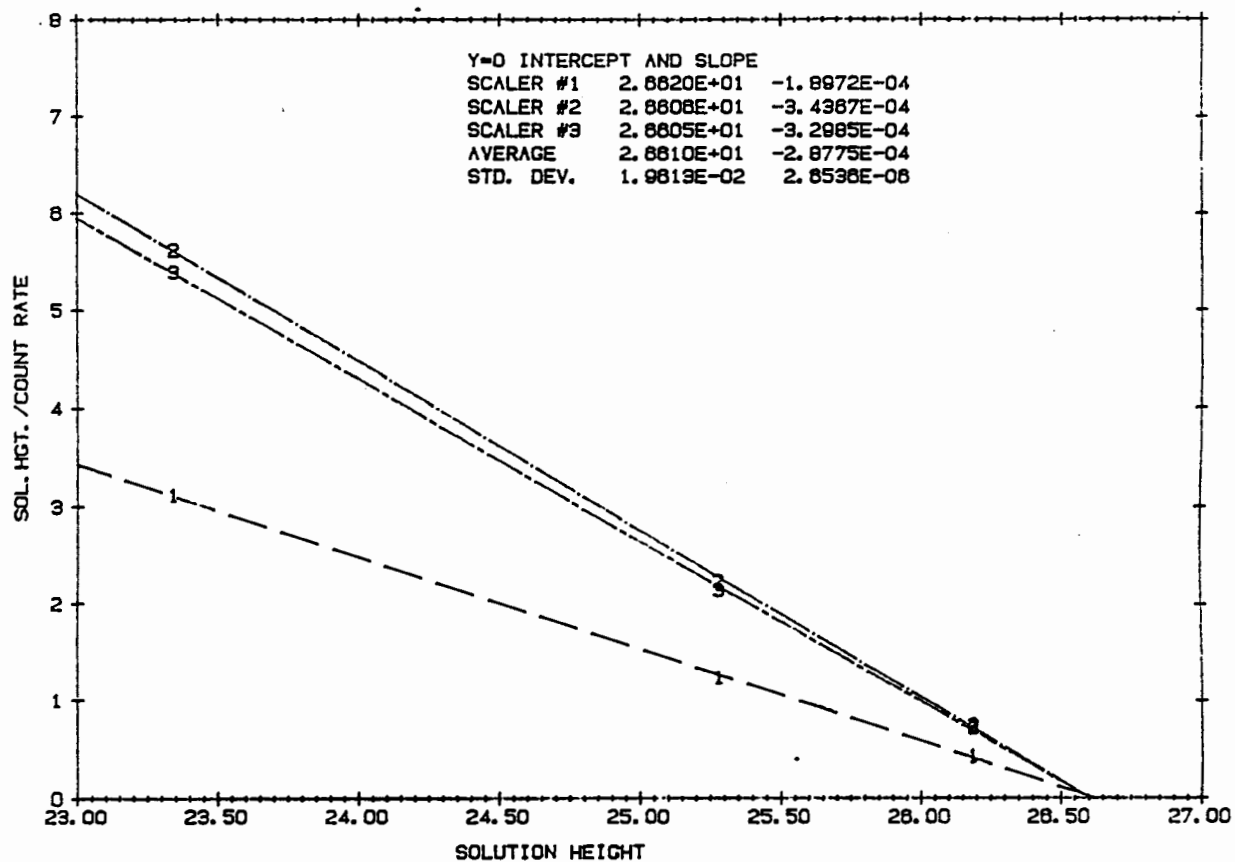


FIGURE C.12 Least Square Fits for CFRP-PNC 074

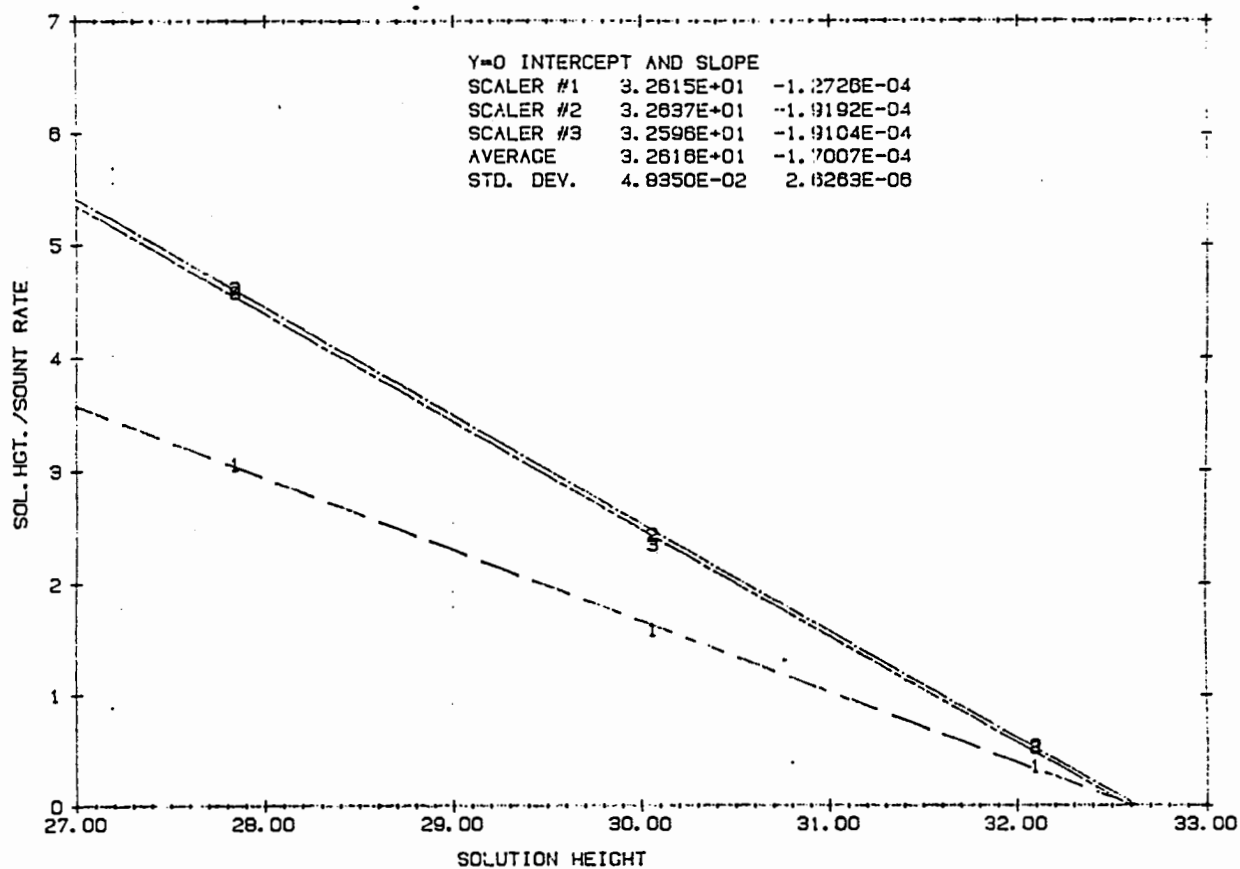


FIGURE C.13 Least Square Fits for CFRP-PNC 075

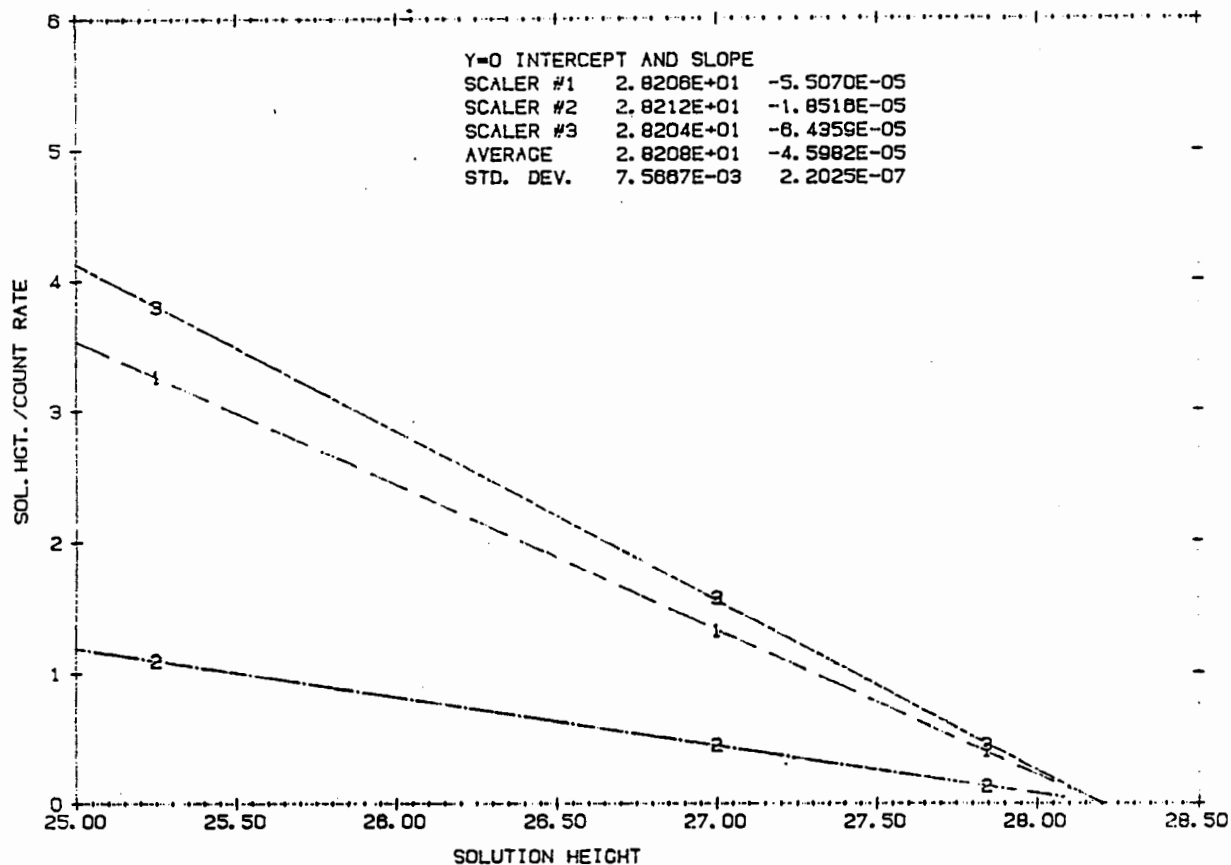


FIGURE C.14 Least Square Fits for CFRP-PNC 076

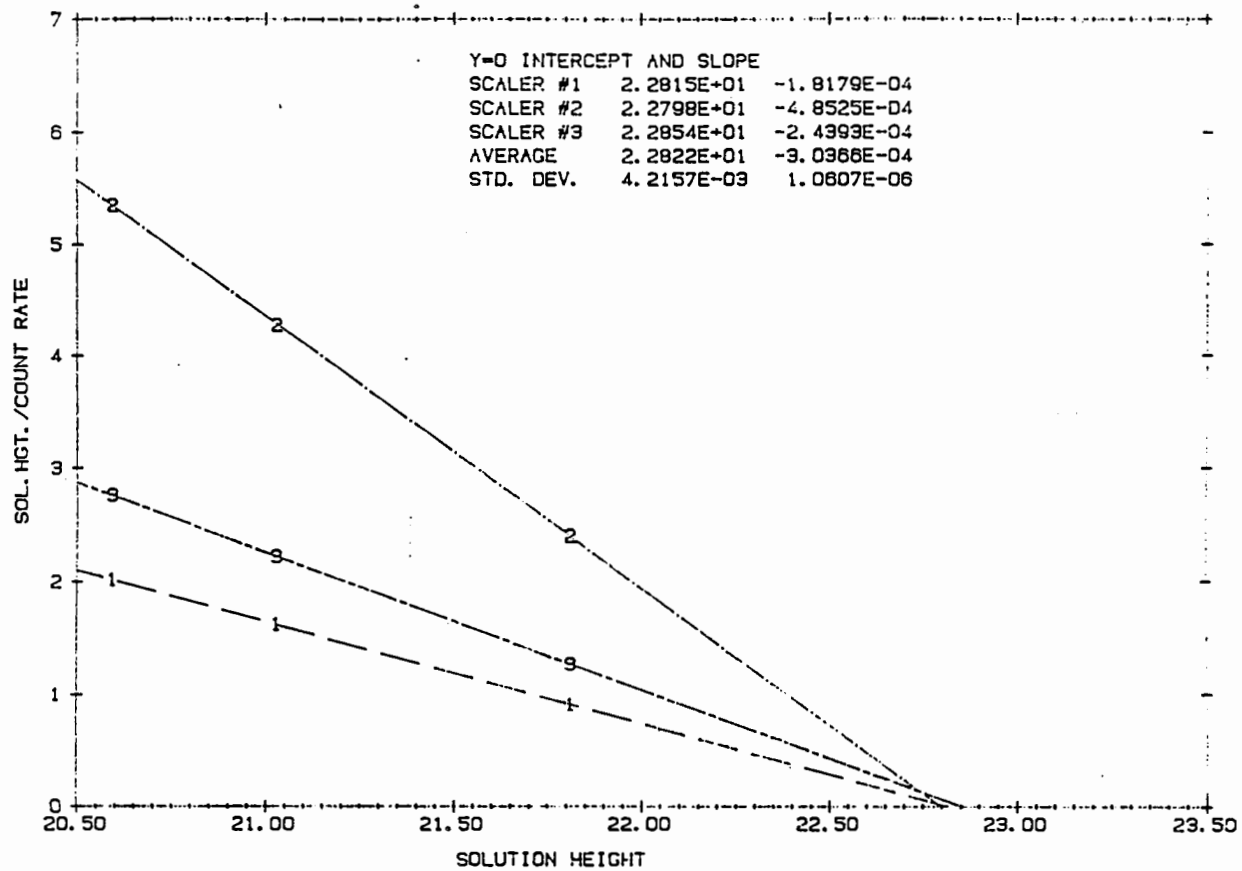


FIGURE C.15 Least Square Fits for CFRP-PNC 077

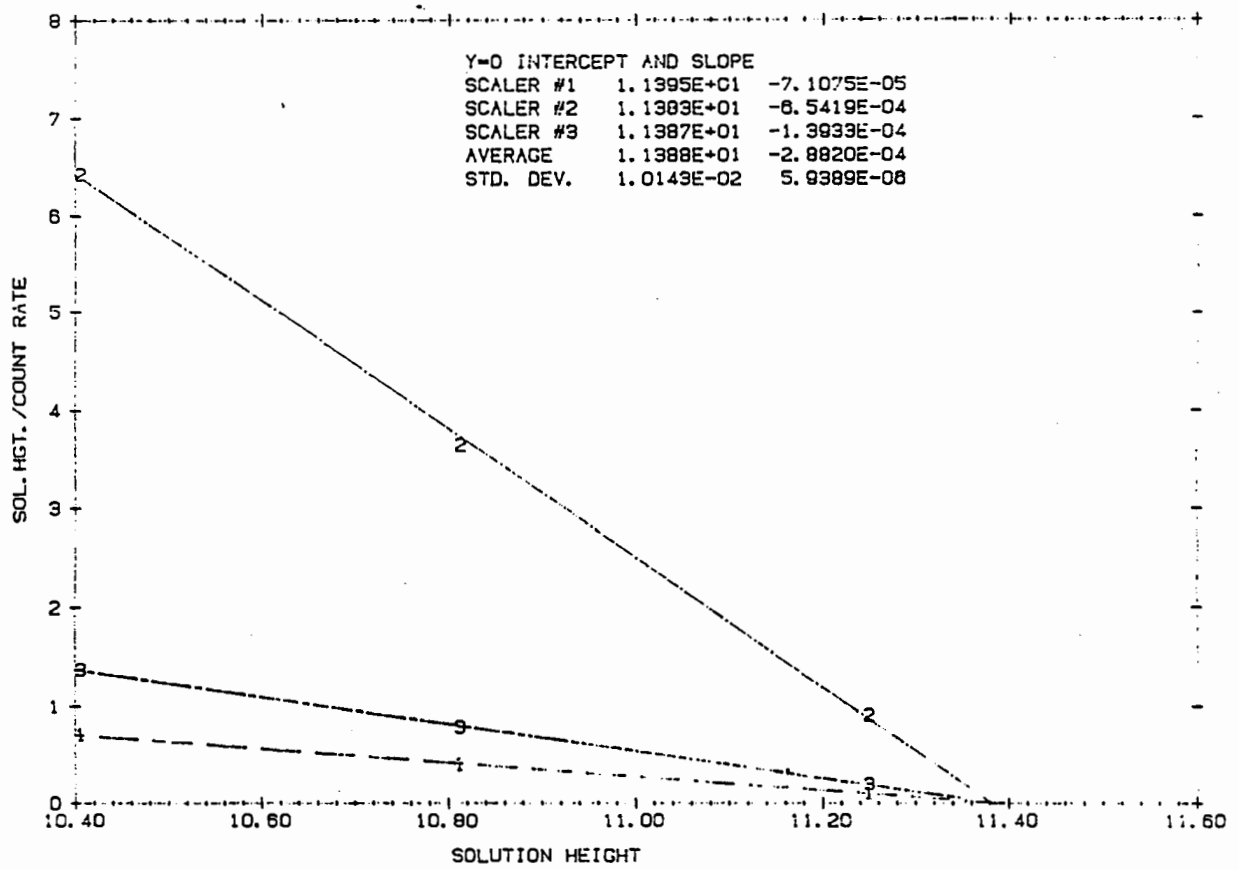


FIGURE C.16 Least Square Fits for CFRP-PNC 078

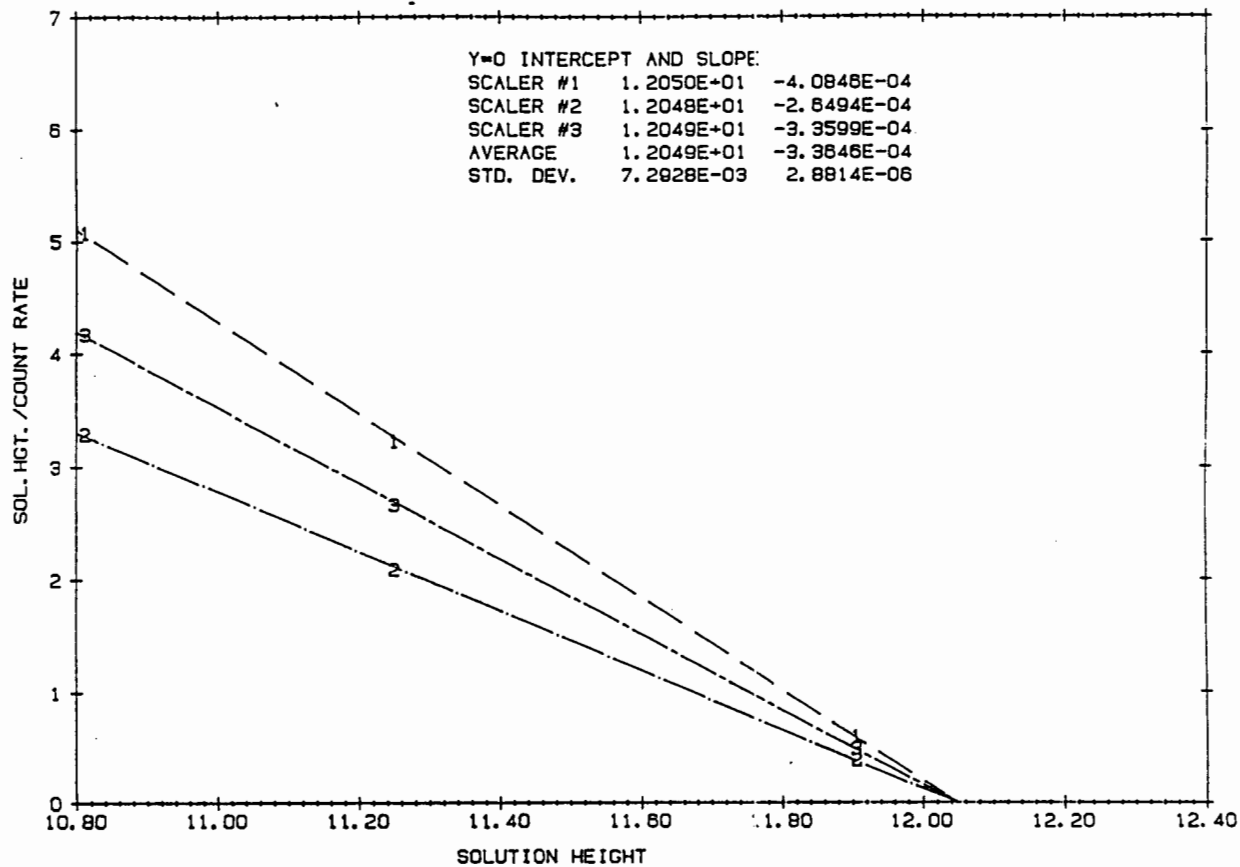


FIGURE C.17 Least Square Fits for CFRP-PNC 083

APPENDIX D

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

APPENDIX D

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

The chemical analyses data for sample 1153 and 1161 are for experiments 063 - 067. The analyses data for 1166 are for experiments 068 - 074. The analyses data for 1170, 1174 and 1177 are for experiments 075 - 078. The analyses data for 1184 are for experiment 083.

The measurement uncertainty for the Spark Source Mass Spectrographic analyses is very large. These measurements are performed primarily for qualitative purposes. The measurement uncertainty for the Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) method is about ± 25 percent.

TABLE D.1 Spectrographic Analysis Report # 1153

LOG NO. <u>#16</u>		Hanford Engineering Development Laboratory		SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT	
MATERIAL <u>mixed oxide</u>				<u>Q6970</u>	
SUBMITTED BY <u>R. LLOYD</u>		SUBMITTER'S NO. <u>1153A</u>		ANALYZED BY <u>JE</u>	
				DATE REPORTED <u>3/12/86</u>	
ELE- MENT		ELE- MENT		ELE- MENT	
Li	<u>20</u>	Ga		Pm	
Be	<u>10</u>	Ge		Sm	
B	<u>10</u>	As		Eu	
F	<u>20</u>	Rb		Gd	
Na	<u>300</u>	Sr		Tb	
Mg	<u>200</u>	Y		Dy	
Al	<u>3000</u>	Zr	<u>1</u>	Ho	
Si	<u>80</u>	Nb		Er	
P	<u>10</u>	Mo		Tm	
S	<u>90</u>	Ru		Yb	
Cl	<u>10</u>	Rh		Lu	
K	<u>40</u>	Pd		Hf	
Ca	<u>500</u>	Ag		Ta	
Sc		Cd		W	
Ti	<u>100</u>	In		Re	
V		Sn		Os	
Cr	<u>1500</u>	Sb		Ir	
Mn	<u>600</u>	Cs		Pt	
Fe	<u>600</u>	Ba		Au	
Co	<u>0.6</u>	La		Hg	
Ni	<u>300</u>	Ce		Tl	
Cu	<u>10</u>	Pr		Pb	
Zn	<u>4</u>	Nd		Bi	
TYPE OF ANALYSIS					
<input type="checkbox"/> QUALITATIVE		<input checked="" type="checkbox"/> SEMIQUANTITATIVE		<input type="checkbox"/> QUANTITATIVE	
<input type="checkbox"/> PARTS PER MILLION		<input checked="" type="checkbox"/> PARTS PER MILLION		<input type="checkbox"/> PARTS PER MILLION	
<input type="checkbox"/> PERCENT		<input type="checkbox"/> PERCENT		<input type="checkbox"/> PERCENT	
<input type="checkbox"/> _____		<input type="checkbox"/> _____		<input type="checkbox"/> _____	
APPR'X PRECISION \pm FACTOR _____		APPR'X PRECISION \pm <u>Factor 3</u>		APPROX PRECISION \pm FACTOR _____	
REMARKS:					
				REPORT APPROVED	

TABLE D.2 Spectrographic Analysis Report # 1161

LOG NO. <u>#18</u>		Hanford Engineering Development Laboratory		SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT	
MATERIAL <u>mixed oxide</u>				<u>Q6697</u>	
SUBMITTED BY <u>E. MURPHY / R. LLOYD</u>		SUBMITTER'S NO. <u>1161A (B)</u>		ANALYZED BY <u>JE</u>	
				DATE REPORTED <u>3/12/86</u>	
ELE- MENT		ELE- MENT		ELE- MENT	
Li	<u>*</u>	Ga		Pm	
Be	<u>3</u>	Ge		Sm	
B	<u>4</u>	As		Eu	
F		Rb		Gd	
Na	<u>300</u>	Sr		Tb	
Mg	<u>70</u>	Y		Dy	
Al	<u>3000</u>	Zr	<u>10</u>	Ho	
Si	<u>80</u>	Nb		Er	
P	<u>300</u>	Mo	<u>2</u>	Tm	
S	<u>100</u>	Ru		Yb	
Cl	<u>20</u>	Rh		Lu	
K	<u>100</u>	Pd		Hf	
Ca	<u>200</u>	Ag		Ta	
Sc		Cd	<u>10</u>	W	
Ti	<u>400</u>	In		Re	
V	<u>1</u>	Sn		Os	
Cr	<u>500</u>	Sb		Ir	
Mn	<u>600</u>	Cs		Pt	
Fe	<u>300</u>	Ba		Au	
Co	<u>2</u>	La		Hg	
Ni	<u>300</u>	Ce		Tl	
Cu	<u>100</u>	Pr		Pb	
Zn	<u>40</u>	Nd		Bi	
TYPE OF ANALYSIS					
<input type="checkbox"/> QUALITATIVE		<input checked="" type="checkbox"/> SEMIQUANTITATIVE		<input type="checkbox"/> QUANTITATIVE	
<input type="checkbox"/> PARTS PER MILLION		<input checked="" type="checkbox"/> PARTS PER MILLION		<input type="checkbox"/> PARTS PER MILLION	
<input type="checkbox"/> PERCENT		<input type="checkbox"/> PERCENT		<input type="checkbox"/> PERCENT	
<input type="checkbox"/> _____		<input type="checkbox"/> _____		<input type="checkbox"/> _____	
APPR'X PRECISION \pm FACTOR _____		APPR'X PRECISION \pm <u>Factor 3</u>		APPROX PRECISION \pm FACTOR _____	
REMARKS:					
				REPORT APPROVED	

BD-7340-021 (4-77)

TABLE D.3 Spectrographic Analysis Report # 1166

LOG NO. <u>21</u>	Hanford Engineering Development Laboratory	SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT	
MATERIAL <u>MIXED OXIDE</u>			
SUBMITTED BY <u>R. LLOYD</u>	SUBMITTER'S NO. <u>1166</u>	ANALYZED BY <u>RT</u>	DATE REPORTED <u>4/15/66</u>

ELE- MENT		ELE- MENT		ELE- MENT	
Li	<u>7</u>	Ga	<u>8</u>	Pm	
Be	<u>*</u>	Ge		Sm	
B	<u>0.4</u>	As		Eu	
F	<u>6</u>	Rb		Gd	
Na	<u>300</u>	Sr		Tb	
Mg		Y		Dy	
Al	<u>300</u>	Zr		Ho	
Si	<u>80</u>	Nb		Er	
P	<u>1</u>	Mo		Tm	
S	<u>30</u>	Ru		Yb	
Cl	<u>1</u>	Rh		Lu	
K	<u>10</u>	Pd		Hf	
Ca	<u>100</u>	Ag	<u>*</u>	Ta	
Sc		Cd		W	
Ti	<u>10</u>	In		Re	
V	<u>1</u>	Sn		Os	
Cr	<u>200</u>	Sb		Ir	
Mn	<u>60</u>	Cs		Pt	
Fe	<u>700</u>	Ba		Au	
Co	<u>0.6</u>	La		Hg	
Ni	<u>30</u>	Ce		Tl	
Cu	<u>4</u>	Pr		Pb	
Zn	<u>1</u>	Nd		Bi	

TYPE OF ANALYSIS		
<input type="checkbox"/> QUALITATIVE <input type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____ APPR'X PRECISION \pm FACTOR _____	<input checked="" type="checkbox"/> SEMIQUANTITATIVE <input checked="" type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____ APPR'X PRECISION \pm <u>FACTOR 3</u>	<input type="checkbox"/> QUANTITATIVE <input type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____ APPROX PRECISION \pm FACTOR _____

REMARKS:

* INTERFERENCE

REPORT APPROVED [Signature]

TABLE D.4 Spectrographic Analyses Reports # 1170 & 1174

LOG NO. <u>#29, #30</u>		Hanford Engineering Development Laboratory		SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT	
MATERIAL <u>mixed oxide</u>					
SUBMITTED BY <u>R. LLOYD</u>		SUBMITTER'S NO.		ANALYZED BY <u>JE</u>	
				DATE REPORTED <u>5/9/86</u>	
ELE- MENT	<u>#29</u>	<u>#30</u>	ELE- MENT	<u>#29</u>	<u>#30</u>
Li	<u>10</u>	<u>10</u>	Ga	<u>80</u>	<u>80</u>
Be	<u>1</u>	<u>1</u>	Ge		
B	<u>1</u>	<u>1</u>	As		
F			Rb		
Na	<u>100</u>	<u>30</u>	Sr		
Mg	<u>10</u>	<u>10</u>	Y		
Al	<u>30</u>	<u>30</u>	Zr	<u>40</u>	<u>40</u>
Si	<u>10</u>	<u>10</u>	Nb		
P	<u>10</u>	<u>3</u>	Mo	<u>1</u>	<u>1</u>
S			Ru		
Cl			Rh		
K	<u>10</u>	<u>4</u>	Pd		
Ca	<u>1</u>	<u>1</u>	Ag	<u>*</u>	<u>*</u>
Sc			Cd	<u>4</u>	<u>4</u>
Ti	<u>40</u>	<u>40</u>	In		
V			Sn	<u>2</u>	<u>2</u>
Cr	<u>200</u>	<u>700</u>	Sb		
Mn	<u>20</u>	<u>20</u>	Cs		
Fe	<u>300</u>	<u>300</u>	Ba		
Co	<u>0.1</u>	<u>0.3</u>	La		
Ni	<u>30</u>	<u>30</u>	Ce		
Cu	<u>100</u>	<u>40</u>	Pr		
Zn	<u>40</u>	<u>10</u>	Nd		
TYPE OF ANALYSIS					
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<input type="checkbox"/> PARTS PER MILLION		<input checked="" type="checkbox"/> PARTS PER MILLION		<input type="checkbox"/> PARTS PER MILLION	
<input type="checkbox"/> PERCENT		<input type="checkbox"/> PERCENT		<input type="checkbox"/> PERCENT	
<input type="checkbox"/> _____		<input type="checkbox"/> _____		<input type="checkbox"/> _____	
APPR'X PRECISION ± FACTOR _____		APPR'X PRECISION ± <u>Factor 3</u>		APPROX PRECISION ± FACTOR _____	
REMARKS:					
<u>#29 = 1170 (Q7056)</u>					
<u>#30 = 1174 (Q7059)</u>					
<u>* Interference</u>					
				REPORT APPROVED <u>Ray</u>	

TABLE D.5 Spectrographic Analysis Report # 1177

LOG NO. <u>33, 34</u>		Hanford Engineering Development Laboratory		SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT	
MATERIAL <u>MIXED OXIDE</u>					
SUBMITTED BY <u>R. LLOYD</u>		SUBMITTER'S NO. <u>SEE BELOW</u>		ANALYZED BY <u>JE & RK</u>	
DATE REPORTED <u>6/3/66</u>					

ELE- MENT			ELE- MENT			ELE- MENT		
Li	33	34	Ga	33	34	Pm	33	34
Be	0.7	70	Ge	<4	<4	Sm	0.6	<0.6
B	*	*	As	2	<2	Eu	0.6	<0.6
F	3	1	Rb			Gd	2	<2
Na	30	30	Sr	20	2	Tb		
Mg	7	7	Y	2	<2	Dy	2	<2
Al	30	30	Zr	40	40	Ho		
Si	8	30	Nb	7	<2	Er		
P	10	10	Mo	6	2	Tm		
S			Ru			Yb		
Cl			Rh			Lu		
K	4	10	Pd			Hf		
Ca	200	200	Ag	*	*	Ta	*	<2
Sc			Cd	1	<1	W	2	<2
Ti	40	50	In			Re		
V	0.3	0.1	Sn	<6	<6	Os		
Cr	200	700	Sb			Ir		
Mn	70	200	Cs	3	<3	Pt		
Fe	300	300	Ba	400	100	Au		
Co	3	1	La	10	30	Hg		
Ni	100	100	Ce	3	<3	Th	150	50
Cu	400	400	Pr			Pb	0.6	0.6
Zn	100	100	Nd			Bi		

TYPE OF ANALYSIS

<input type="checkbox"/> QUALITATIVE <input type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____	<input checked="" type="checkbox"/> SEMIQUANTITATIVE <input checked="" type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____	<input type="checkbox"/> QUANTITATIVE <input type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____
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APPR'X PRECISION ± FACTOR _____ APPR'X PRECISION ± FACTOR 3 APPROX PRECISION ± FACTOR _____

REMARKS:

33 = 1152 C-

34 = 1177 A

REPORT APPROVED [Signature]

TABLE D.6 Spectrographic Analysis Report # 1184

LOG NO. _____		Hanford Engineering Development Laboratory		SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT	
MATERIAL Mixed Oxide					
SUBMITTED BY E MURPHY		SUBMITTER'S NO. below		ANALYZED BY JE	
DATE REPORTED 7/1/86					

ELE- MENT	43	44	ELE- MENT	43	44	ELE- MENT	43	44
Li	0.2	0.7	Ga	20	20	Pm		
Be	10	4	Ge	40		Sm		
B	3	1	As			Eu		
F			Rb			Gd	2	
Na	30	30	Sr	10	10	Tb		
Mg	10	10	Y	20		Dy		
Al	100	100	Zr	40	4	Ho		
Si	80	30	Nb	2		Er		
P	100	100	Mo	90	30	Tm		
S			Ru			Yb		
Cl			Rh			Lu		
K	10	10	Pd			Hf		
Ca	200	200	Ag	*	*	Tl	50	50
Sc			Cd	40	4	W		
Ti	100	10	In			Re		
V	1	0.3	Sn	10	10	Os		
Cr	200	70	Sb	40	40	Ir		
Mn	70	20	Cs			Pt		
Fe	1000	300	Ba	100	100	Au		
Co	1	0.3	La			Hg		
Ni	100	30	Ce			Tl	20	70
Cu	100	40	Pr			Pb	6	2
Zn	100	40	Nd			Bi		

TYPE OF ANALYSIS

<input type="checkbox"/> QUALITATIVE <input type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____	<input checked="" type="checkbox"/> SEMIQUANTITATIVE <input checked="" type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____	<input type="checkbox"/> QUANTITATIVE <input type="checkbox"/> PARTS PER MILLION <input type="checkbox"/> PERCENT <input type="checkbox"/> _____
APPR'X PRECISION ± FACTOR _____	APPR'X PRECISION ± Factor 3	APPROX PRECISION ± FACTOR _____

REMARKS:

43 = 1184 A

44 = 1185 A

* Interference

REPORT APPROVED Raj

TABLE D.7 Inductively Coupled Plasma
Spectroscopy Analysis (mg/liter)

<u>Element</u>	<u>Sample Number 1153</u>
Al	33.8
B	8.6
Ba	2.6
Ca	24.3
Cd	2.7
Ce	8.9
Cr	28.8
Cu	5.2
Dy	8.3
Fe	72.7
Gd	<1
K	3.8
La	8.3
Li	8.2
Mg	6.6
Mn	5.6
Mo	8.5
Na	22.6
Nd	8.6
Ni	28.8
Rh	2.4
Ru	1.2
Si	28.6
Sr	8.1
Te	(a)
Ti	3.7
Zn	<1
Zr	8.4

(a) Element not detected.

APPENDIX E

CHEMICAL ANALYSES DATA OF THE REFLECTOR WATER SAMPLES

TABLE E.1 Water Samples Analyses - 064 & 066



HANFORD ENVIRONMENTAL
HEALTH FOUNDATION

CO 10311

April 3, 1986

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

Attn: Ray Lloyd

WATER SAMPLES ANALYSES

The results of the three water samples received February 18, 1986, are following. Analyses were done in accordance with Standard Methods for the Analysis of Water and Wastewater, 16th Edition.

Parameter	Samples		
	062	064	066
pH	7.4	7.5	7.5
Total alkalinity mg/L	53.8	54.7	55.2
HCO ₃ Alkalinity mg/L	51	52	52
CO ₃ Alkalinity mg/L	<0.5	<0.5	<0.5
Total dissolved solids mg/L	82	88	86
Sulfate mg/L	17.9	17.2	17.3
Nitrate-N mg/L	0.12	0.10	0.12
Chloride mg/L	2.1	2.0	3.4
Fluoride mg/L	0.10	0.10	0.15
Cadmium mg/L	<0.0005	<0.0005	<0.0005
Copper mg/L	<0.05	<0.05	<0.05
Chromium mg/L	<0.005	<0.005	<0.005
Iron mg/L	0.06	0.08	0.26
Lead mg/L	<0.005	<0.005	<0.005
Manganese mg/L	<0.01	<0.01	<0.01
Zinc mg/L	<0.05	0.26	0.66

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

P. A. Thurman

P. A. Thurman
Environmental Health Sciences

jt

TABLE E.2 Water Sample Analyses - 069, 071 & 072



HANFORD ENVIRONMENTAL
HEALTH FOUNDATION

June 12, 1986

CO-10507

Pacific Northwest Laboratory
209-E
200-E Area

Attn: R. Lloyd

WATER SAMPLES ANALYSES

Results of the analyses of the 2 water samples received March 21, 1986, are following. Analyses were done in accordance with Standard Methods for the Examination of Water and Wastewater, 16th Ed.

Parameter	Results	
	069	071-072
pH	7.4	8.0
Total Alkalinity	56.6 mg/L	52.8 mg/L
Alkalinity-carbonate	<0.5 mg/L	0.6 mg/L
Alkalinity-bicarbonate	56 mg/L	50 mg/L
Total dissolved solids	91 mg/L	70 mg/L
Fluoride	<0.1 mg/L	<0.1 mg/L
Chloride	2.4 mg/L	2.7 mg/L
Nitrate	0.41 mg/L	0.36 mg/L
Sulfate	16 mg/L	15 mg/L
Lead	<0.005 mg/L	<0.005 mg/L
Cadmium	<0.0005 mg/L	<0.0005 mg/L
Copper	<0.05 mg/L	<0.05 mg/L
Chromium	0.013 mg/L	<0.005 mg/L
Iron	0.07 mg/L	0.38 mg/L
Manganese	0.014 mg/L	0.012 mg/L
Zinc	0.74 mg/L	0.54 mg/L

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

P. A. Thurman

P. A. Thurman
Environmental Health Sciences

kw

TABLE E.3 Water Sample Analyses - 076 & 078



HANFORD ENVIRONMENTAL
HEALTH FOUNDATION

Parameter	Sample Results	
	076	078
pH	7.2	7.8
Total Alkalinity mg/L	43.5	42.1
HCO ₃ Alkalinity mg/L	42.5	41.0
CO ₃ Alkalinity mg/L	<0.5	<0.5
Total Dissolved Solids mg/L	104	93
Sulfate mg/L	13.8	14.6
Nitrate (as N) mg/L	<0.05	<0.05
Chloride mg/L	3.1	2.5
Fluoride mg/L	<0.1	<0.1
Iron mg/L	0.11	0.31
Cadmium mg/L	<0.0005	<0.0005
Copper mg/L	<0.05	<0.05
Chromium mg/L	<0.005	<0.005
Lead mg/L	<0.05	<0.05
Manganese mg/L	0.01	0.02
Zinc mg/L	0.53	0.40

APPENDIX F

COMPOSITION OF THE CONCRETE REFLECTOR

APPENDIX F

COMPOSITION OF THE CONCRETE REFLECTOR^(a)

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 the data are 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 $[1/\Sigma(1/\sigma_i^2)]^{1/2}$.

The water content was determined by the following procedure. Two concrete samples were separated into two aliquots. Each of the samples was weighed, then heated to 100°C for 1 hours, cooled, and reweighed. The samples were then heated to 1000°C 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 was determined to be 1.05 ± 0.03 weight percent.

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

TABLE F.1 Concentration of Elements in Concrete Reflector^(a)

Element	Unit of Measure (Wt)	INAA ^(b)		XRF ^(c)		Average
		1	2	1	2	
Na	%	1.40 ± 0.01	1.46 ± 0.01	-	-	1.43 ± 0.007
Mg	%	0.64 ± 0.46	1.00 ± 0.50	-	-	0.92 ± 0.34
Al	%	5.27 ± 0.03	5.58 ± 0.03	4.0 ± 1.4	4.3 ± 1.4	4.79 ± 0.02
Si	%	-	-	22.3 ± 1.5	23.9 ± 1.6	23.1 ± 1.1
P	%	-	-	<0.6	<0.6	<0.6
S	%	-	-	0.40 ± 0.09	0.36 ± 0.09	0.38 ± 0.06
K	%	0.86 ± 0.14	0.70 ± 0.15	0.67 ± 0.04	0.67 ± 0.04	0.72 ± 0.03
Ca	%	10.8 ± 0.5	10.4 ± 0.5	13.8 ± 0.7	12.9 ± 0.7	12.0 ± 0.3
Ti	ppm	3910 ± 340	3770 ± 340	2900 ± 200	2700 ± 200	3320 ± 122
V	ppm	91.3 ± 3.6	85.5 ± 3.8	91 ± 31	140 ± 30	102 ± 3
Cr	ppm	-	-	196 ± 21	134 ± 19	165 ± 14
Mn	ppm	557 ± 4	520 ± 4	606 ± 36	577 ± 34	565 ± 3
Fe	%	-	-	3.45 ± 0.17	3.29 ± 0.16	3.37 ± 0.12
Co	ppm	-	-	<49	<49	<49
Ni	ppm	-	-	57 ± 4	52 ± 6	54 ± 3
Cu	ppm	-	-	42 ± 3	39 ± 3	40 ± 2
Zn	ppm	-	-	176 ± 9	146 ± 8	161 ± 6
Ga	ppm	-	-	12 ± 1	11 ± 1	11.5 ± 0.7
Pb	ppm	-	-	34 ± 2	23 ± 2	28 ± 1
As	ppm	-	-	33 ± 2	29 ± 2	31 ± 1
Rb	ppm	-	-	21 ± 1	20 ± 1	20.5 ± 0.7
Sr	ppm	-	-	200 ± 20	300 ± 30	290 ± 17
Y	ppm	-	-	15 ± 1	15 ± 1	15 ± 0.7
Zr	ppm	-	-	85 ± 7	87 ± 7	86 ± 5
Nb	ppm	-	-	5.4 ± 1.0	5.3 ± 0.7	5.4 ± 0.6
Mo	ppm	-	-	6.9 ± 0.8	5.6 ± 0.8	6.2 ± 0.6

(a) Reported error is the 1 σ value for each sample. The weighted standard deviation is reported for the average.

(b) Isotope neutron activation analysis method.

(c) X-ray fluorescence method.

TABLE F.2 Water Content of Concrete Reflector

Sample Number	Weight Loss After Heating to 100°C (wt%) (a)	Weight Loss After Heating to 1000°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

(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 $2.3 \pm 0.1 \text{ g/cm}^3$. The computed atom densities for each element are also listed in Table F.3.

TABLE F.3 Calculated Atom Densities for the Concrete Reflector

Element	Weight Percent	Atom Density (Atoms/b.cm)
O	51.91	4.553E-2
Si	23.10	1.154E-2
Ca	12.00	4.201E-3
Al	4.79	2.491E-3
Fe	3.37	8.468E-4
Na	1.43	8.728E-4
H	1.05	1.462E-2
Mg	0.92	5.310E-4
K	0.72	2.584E-4
S	0.38	1.663E-4
Ti	0.33	9.667E-5

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