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# Criticality Experiments with Mixed Oxide Fuel Pin Arrays in Plutonium-Uranium Nitrate Solution

R. C. Lloyd  
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August 1988

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

Pacific Northwest Laboratory  
Operated for the U.S. Department of Energy  
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PNL-6559

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CRITICALITY EXPERIMENTS WITH MIXED OXIDE FUEL PIN  
ARRAYS IN PLUTONIUM-URANIUM NITRATE SOLUTION

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and

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Fuel Reprocessing Program and the  
Power Reactor and Nuclear Fuel Development  
Corporation of Japan under the Joint  
Memorandum of Agreement on Criticality  
Data Development

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## SUMMARY

A series of critical experiments was completed with mixed plutonium-uranium solutions having a Pu/(Pu + U) ratio of approximately 0.22 in a boiler tube-type lattice assembly. These experiments were conducted as 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 the experiments and data are included in this report. The experiments were performed with an array of mixed oxide fuel pins in aqueous plutonium-uranium solutions. The fuel pins were contained in a boiler tube-type tank and arranged in a 1.4 cm square pitch array which resembled cylindrical geometry. One experiment was performed with the fuel pins removed from the vessel. The experiments were performed with a water reflector. The concentration of the solutions in the boiler tube-type tank was varied from 4 to 468 g (Pu + U)/liter. The ratio of plutonium to total heavy metal (plutonium plus uranium) was approximately 0.22 for all experiments.

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# CRITICALITY EXPERIMENTS WITH MIXED OXIDE FUEL PIN ARRAYS IN PLUTONIUM-URANIUM NITRATE SOLUTION

## 1.0 INTRODUCTION

The design and operation of facilities for recycling Fast Breeder Reactor (FBR) fuels involve 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 such nuclear criticality aspects as they related to the development of FBR fuel recycling 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 Program, 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.

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, result in uncertainties on the particular conditions for FBR fuel and need further verification based on actual critical experimental data. Therefore, experimental data are needed which will allow verification 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. These data have application whenever mixtures of plutonium and uranium exist, such as in the head-end dissolution process of a fuel reprocessing plant and wherever heterogeneous systems exist.

This report contains a description of the criticality experiments and the data from the experiments. The experiments were conducted with mixed plutonium-uranium solutions at a Pu/(Pu + U) ratio of approximately 0.22. The experiments were performed in the boiler tube-type lattice assembly which contained an array of mixed oxide fuel pins. The measurements were made with a water reflector. The concentration of the solution in the boiler tube-type tank was varied from 4 to 468 g (Pu + U)/liter. Experiments were also performed with gadolinium added to the solution to determine its effectiveness as a neutron poison in a mixed oxide fuel pin array.

## 2.0 DESCRIPTION OF EXPERIMENTAL ASSEMBLIES

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

### 2.1 GENERAL DESCRIPTION OF THE SOLUTION SYSTEM

An existing experimental system, previously used for solution experiments at the Critical Mass Laboratory, was used for conducting the measurements and providing 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. The cylindrical vessel assembly positioned south of Hood 1 will be discussed in Section 2.2.

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, when desired. The boiler tube-type vessel was used in this series of experiments.

The fill, dump and manometer lines enter the bottom of the vessel through the dump valve system. The dump valve and lines are designed to drain the fissile solution even if the addition were continued. The vessel is connected to the dump valve pedestal by a Marmon flange connection which provides a leak tight connection.

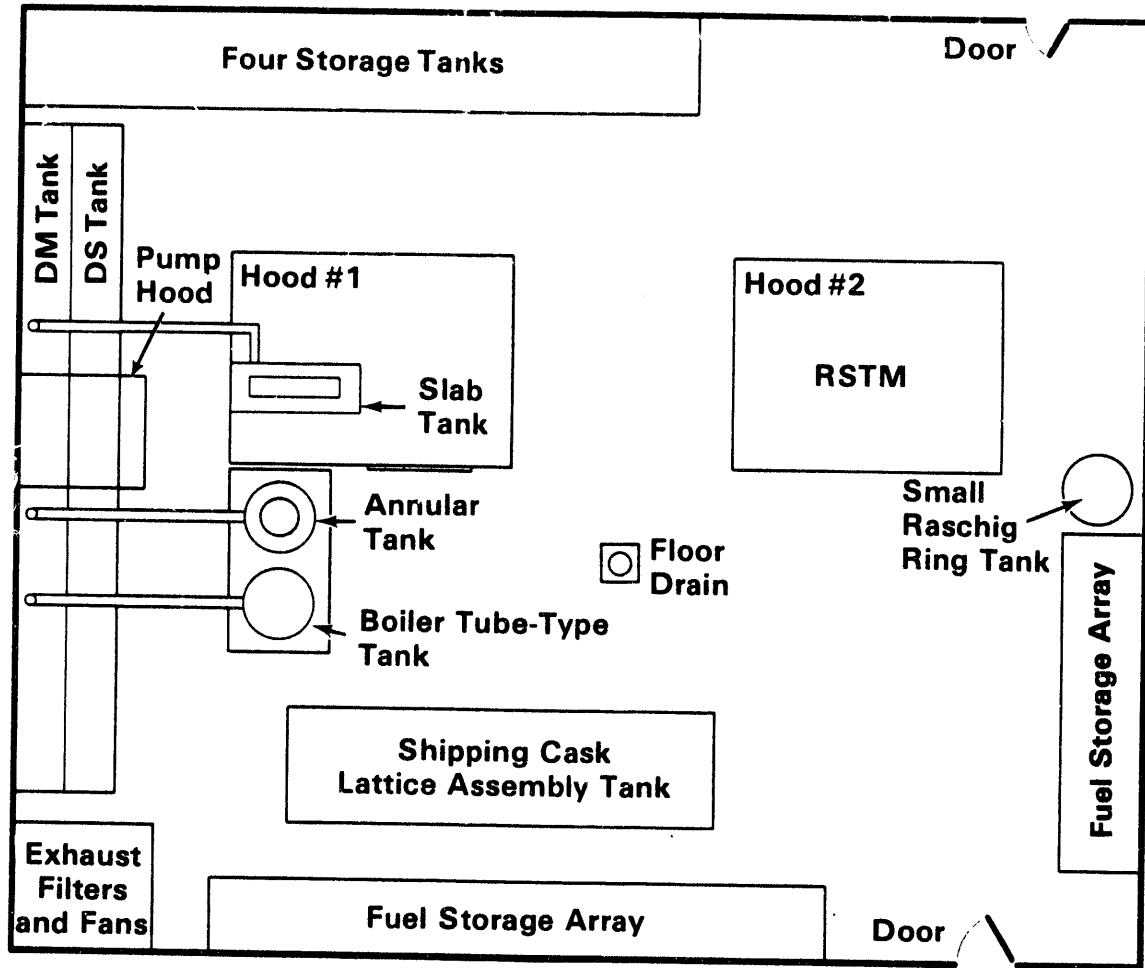
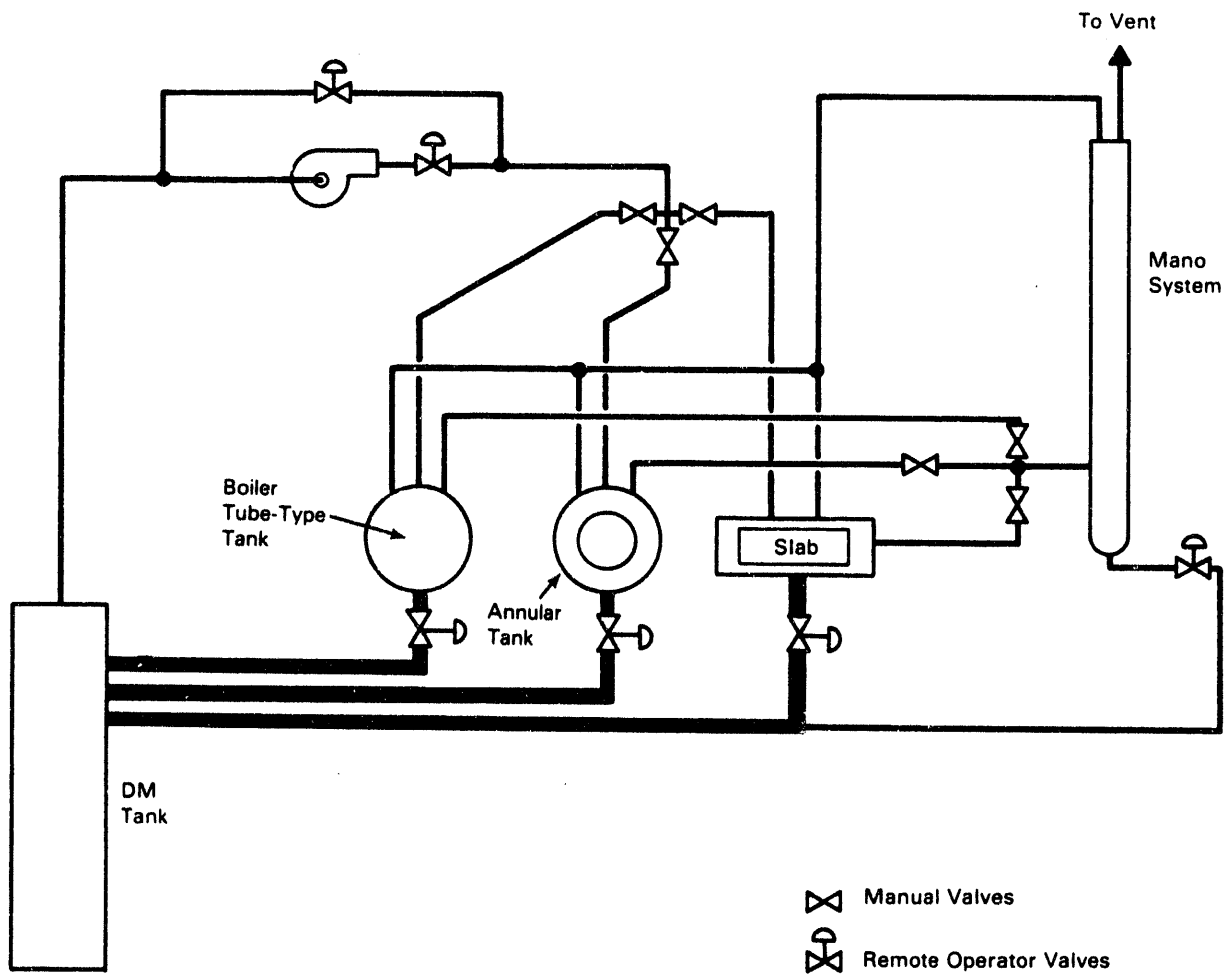


FIGURE 2.1 Floor Plan Schematic of the Critical Assembly Room



**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 vessel used for the experiments discussed in this report was a boiler tube-type tank and it was located on the left side of the system. The control and safety blade mechanisms were mounted above the vessel and can be seen in the figure. The reflector tank serves to contain water for the water-reflected experiments. Windows of polycarbonate (Lexan) were installed on the front for access to the experimental vessels and for visual inspection. The reflector tank was fabricated of carbon steel. A schematic of the experimental vessel arrangement giving dimensions is shown in Figure 2.4.

A boiler tube-type tank was fabricated to provide a fixed array of guide tubes. During critical experiments, 996 Type 3.2 Fast Flux Test Facility (FFTF) fuel pins were placed inside the tubes, and the tubes were surrounded by various solutions. A schematic of the vessel is shown in Figure 2.5. The vessel has an outside diameter of 53.188 cm. The wall thickness of the vessel is 0.091 cm. This particular design of the vessel was necessary in order to isolate the fuel pins from plutonium-bearing solutions. The design of the vessel incorporated a means of attaching the guide tubes to the top plate in a manner that did not warp the plate. The tubes were sealed by 0.635 cm long plugs laser welded into the ends of the tubes. The other end of the tube was flared and mechanically sealed with a screw-type compression fitting. Only the mechanically sealed end of the tubes were attached to the top plate to prevent bowing of the tubes as a result of differential thermal expansion. The thickness of the top plate is 1.930 cm. An elevation view of the boiler tube-type tank is shown in Figure 2.6.

The vessel also included a middle lattice plate and a polyethylene spacer to maintain a uniform spacing between tubes throughout the vessel. The diameter and thickness of the middle lattice plate is 52.426 and 0.478 cm, respectively. The diameter of the holes drilled in the plate for the guide tubes is  $0.675 \pm 0.004$  cm.

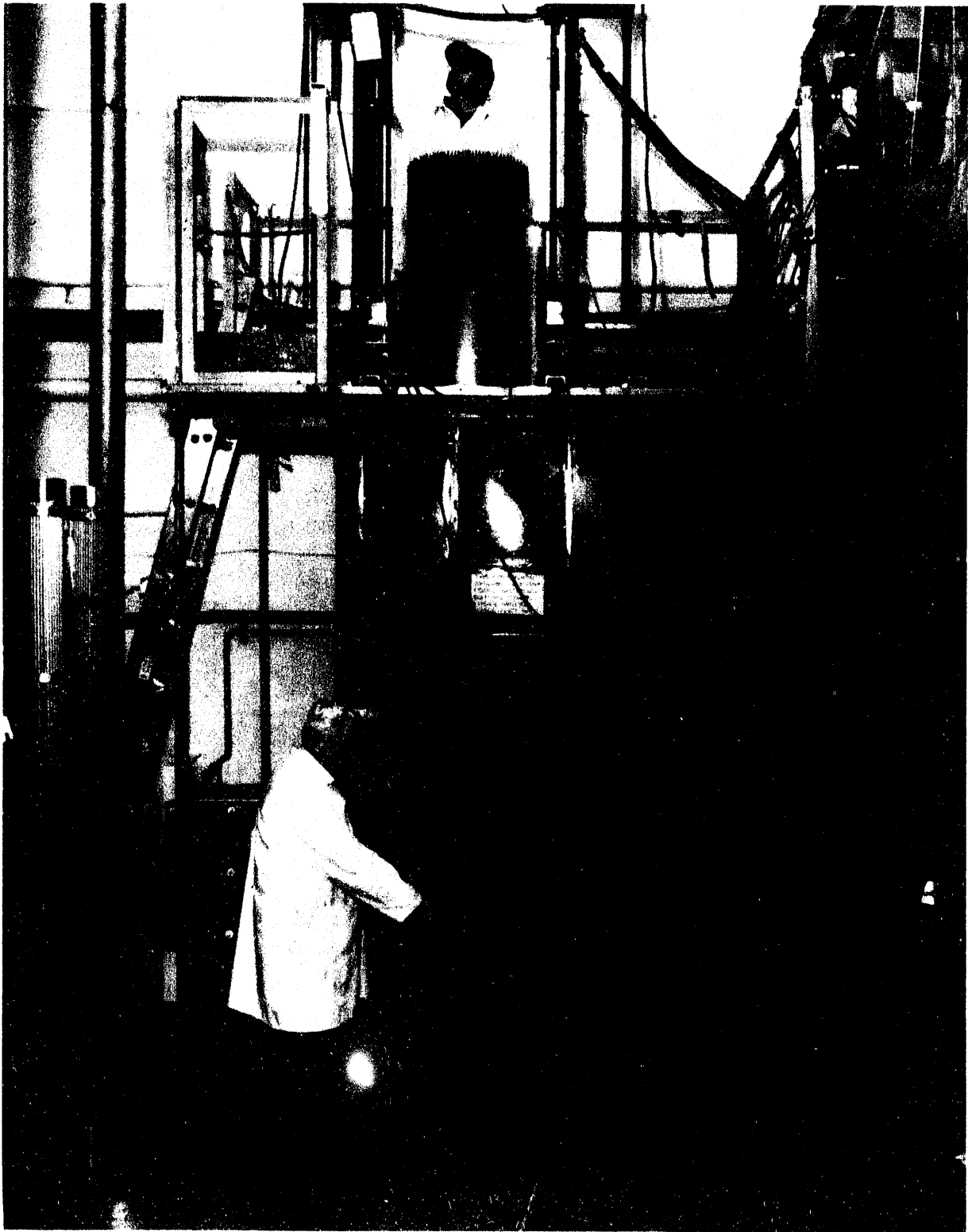
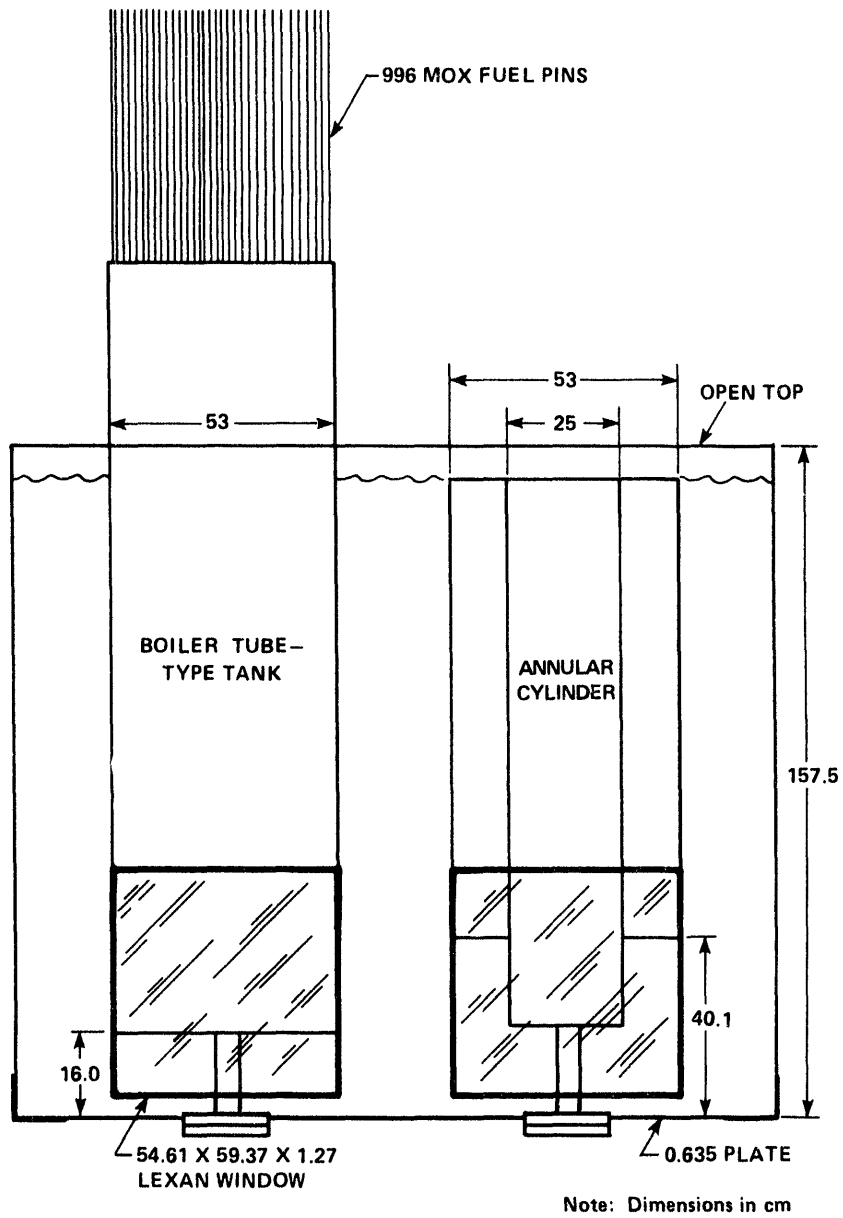


FIGURE 2.3 Photograph of the Cylindrical Vessel System



**FIGURE 2.4** Schematic of Dual Cylinder Tank with Boiler Tube-Type Tank and Annular Cylinder Mounted



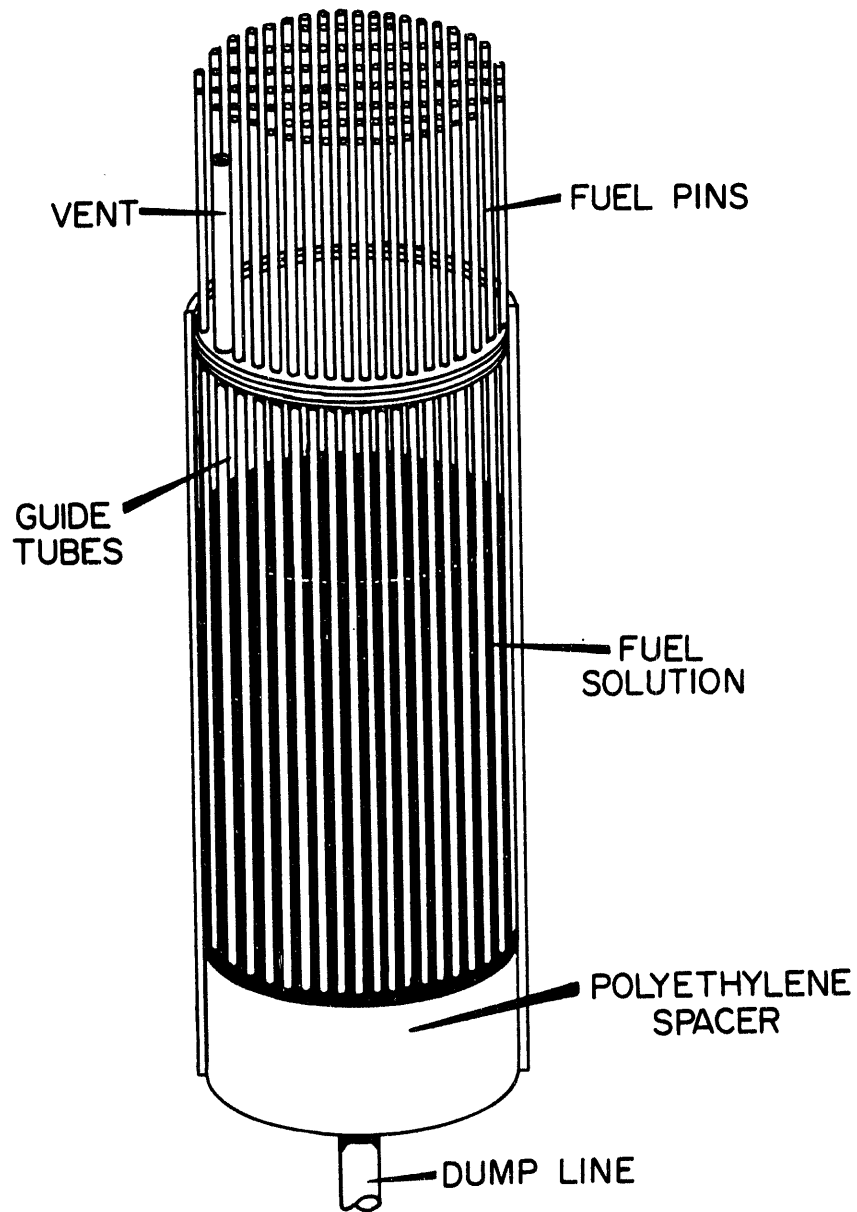


FIGURE 2.5 Schematic of Boiler Tube-Type Tank

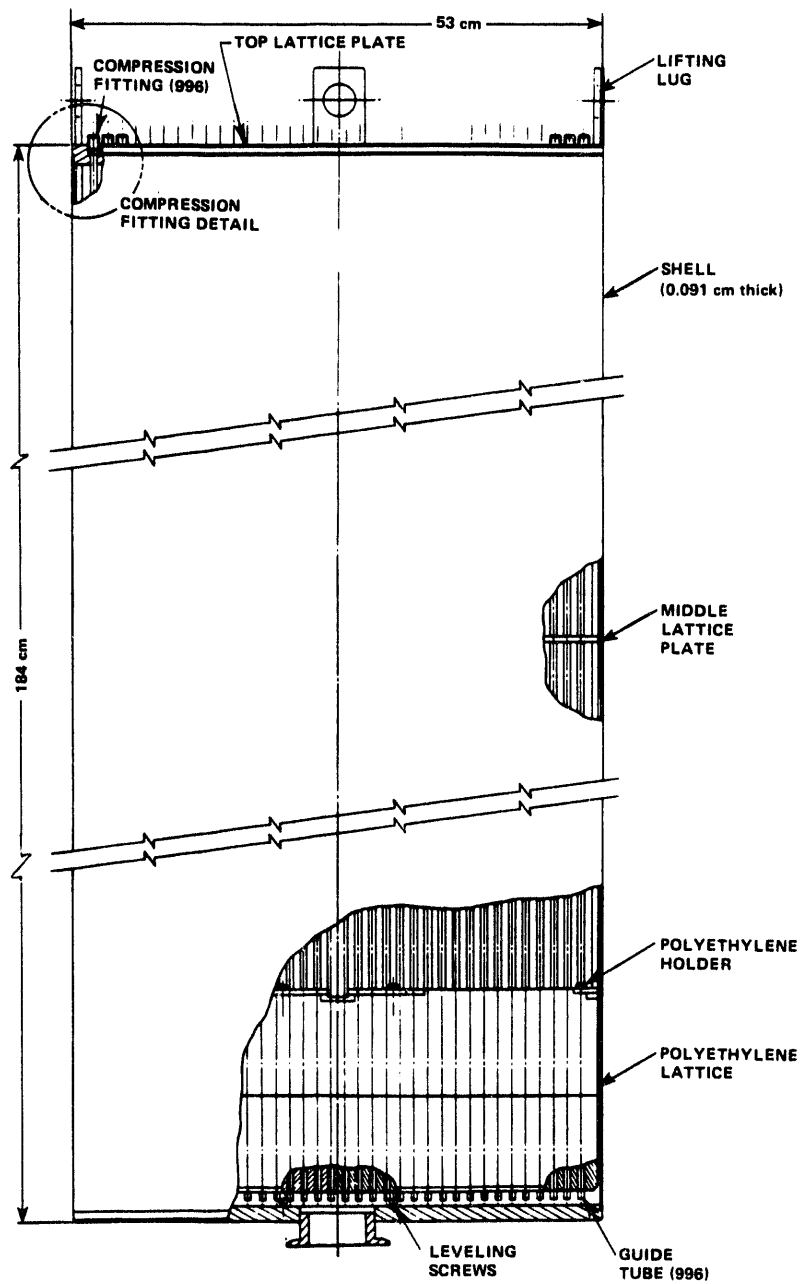


FIGURE 2.6 Elevation View of Boiler Tube-Type Tank

The polyethylene spacer that was placed at the bottom of the vessel, in the region below the mixed oxide (MOX) fuel, serves to effectively exclude fissile solution from this region. Therefore, neutron multiplication occurs only in the region of interest (i.e., where the MOX is surrounded by fissile solution). The diameter of the 996 holes in the polyethylene spacer is  $0.701 \pm 0.0076$  cm. The diameter and height of the spacer is 51.511 and 21.184 cm, respectively. Figure 2.7 is a photograph of the polyethylene spacer which shows that the spacer was fabricated from three separate pieces of polyethylene that were held together by tie bolts. The four pairs of tie bolts were secured to the four spacer holders. The middle lattice plate and the polyethylene spacer were suspended inside the vessel by four rectangular bars (cross sectional dimensions are 0.635 by 1.905 cm). In Figure 2.8, the photograph shows the inner assembly and guide tubes used in as-built dimension measurements. The portion of the assembly where the MOX was located, between the middle lattice plate and the spacer, is indicated.

The vessel was designed so that the bottom of MOX fuel would be at the same elevation as the upper surface of the polyethylene spacer. Based on measurements made after fabrication of the inner assembly, the top surface of the spacer was estimated to be 0.028 cm above the bottom of the MOX fuel. However, since the uncertainty in these measurements is 0.127 cm, the levels should be considered to have the same elevation. Also, the bottom of the guide tube plugs was found to extend beyond the spacer by  $0.04 \pm 0.15$  cm. The uncertainties associated with the above two measured values are large because these measurements were not made directly. Instead, these values were determined by taking the difference between a series of measurements taken inside and outside selected guide tubes. The spacer is 0.922 cm above the inside surface of the bottom plate of the vessel to allow for filling and draining. Four set screws were inserted into the spacer to assure that the vessel could be drained.

An analysis of the top plate, middle lattice plate and polyethylene spacer true position measurement data found that the holes had a square pitch of  $1.400 \pm 0.003$  cm. In Figure 2.9, the photograph is a view under the polyethylene spacer showing the plug-end of the 996 guide tubes and the

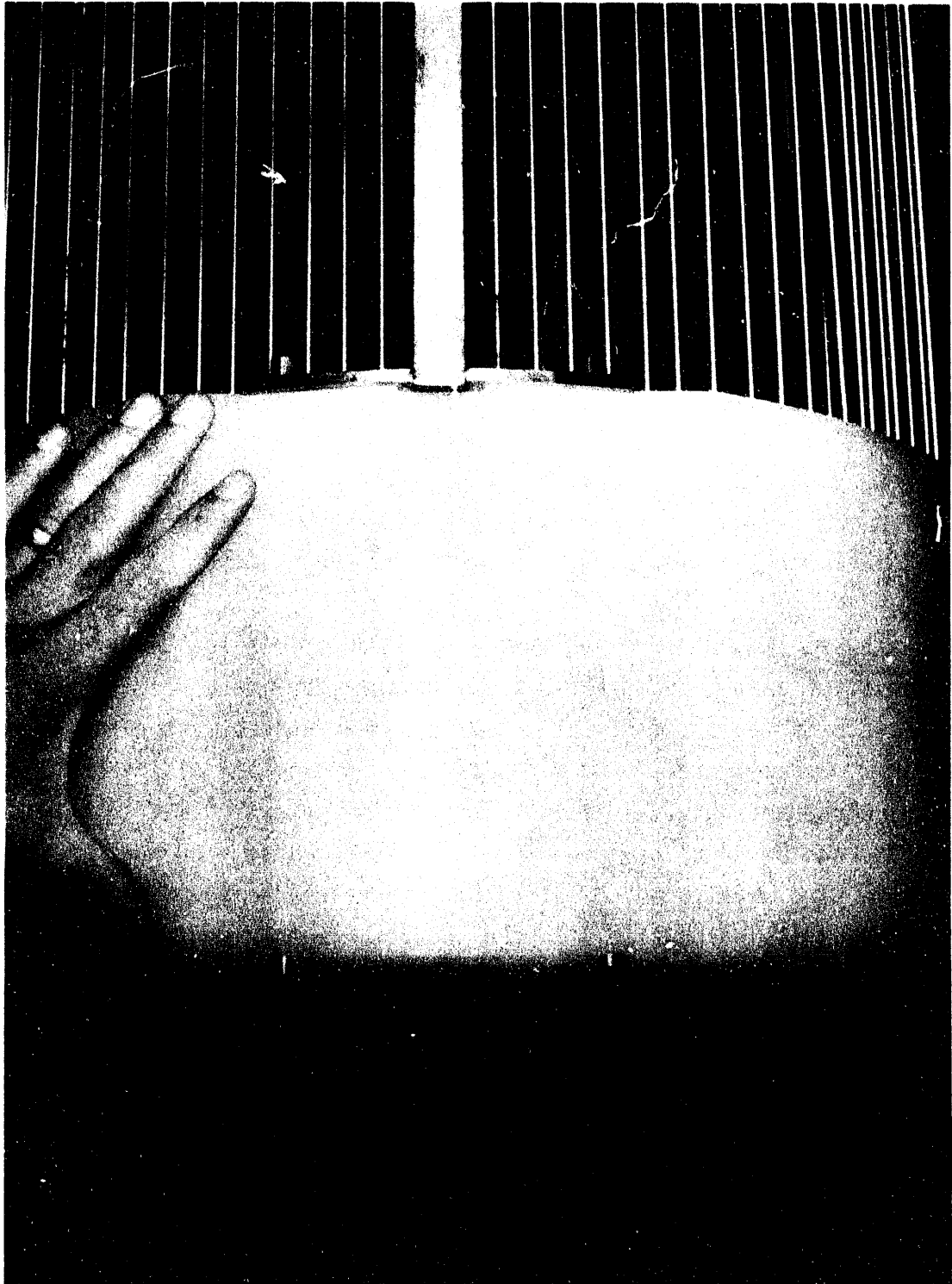


FIGURE 2.7 Photograph of Polyethylene Spacer



FIGURE 2.8 Photograph of Boiler Tube-Type Tank Inner Assembly

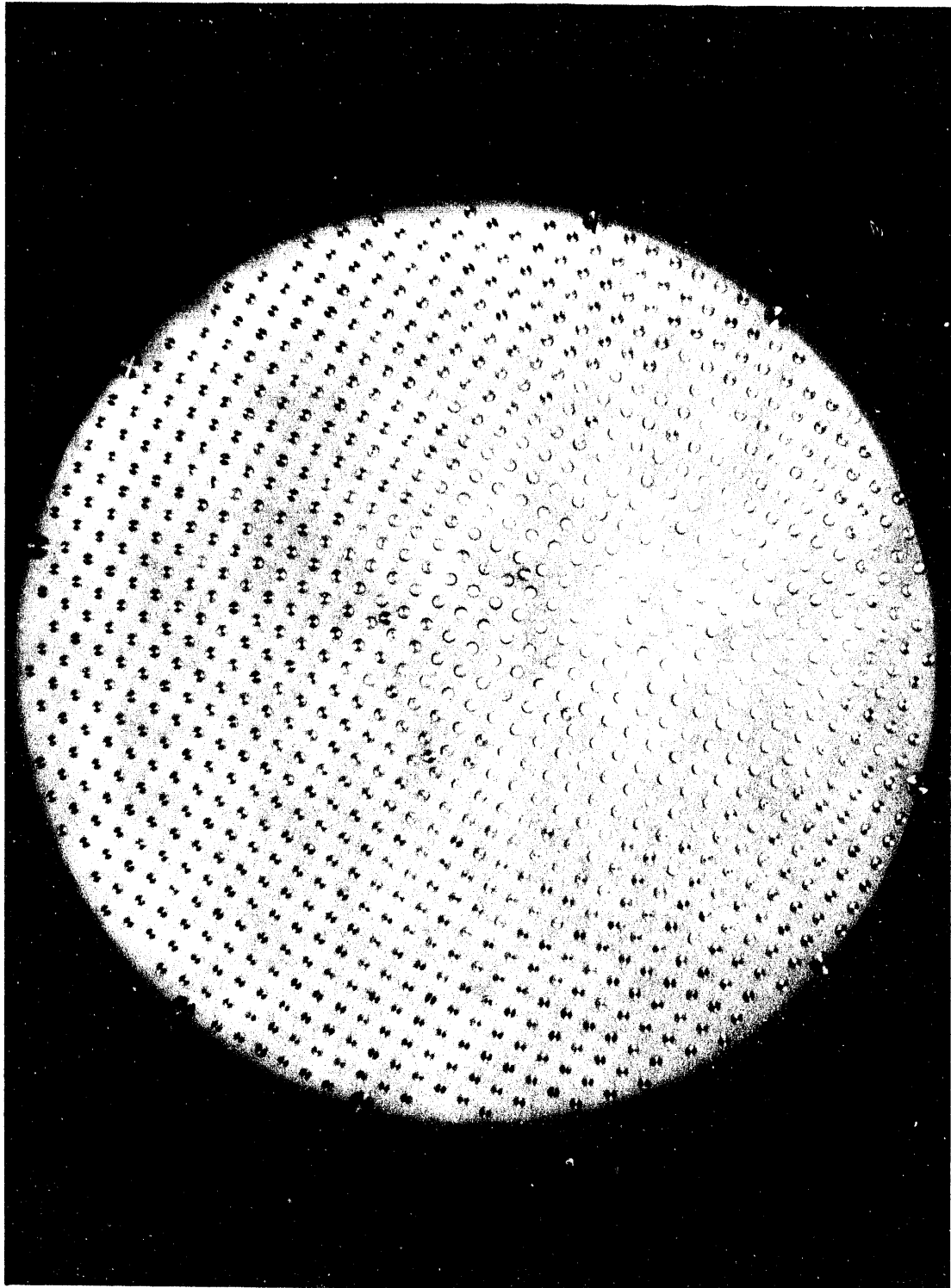


FIGURE 2.9 Photograph of Polyethylene Spacer Showing Lattice Configuration

geometrical arrangement of the tubes. Also shown are the four leveling screws and the eight tie bolts. The completed inner assembly is shown in Figure 2.10, and the completed boiler tube-type tank is shown in Figure 2.11. The schematic of the vessel illustrated in Figure 2.12 summarizes all of the vital as-built dimensions.

All materials used in the fabrication of the boiler tube-type tank were certified Type 304L stainless steel, except for the compression fittings (Type 17-4PH stainless steel) and the polyethylene spacer. The spacer was fabricated from blocks of Number 213 "pure polyethylene" supplied by Reactor Experiments, Inc. The listed density for this material is  $0.9 \text{ g/cm}^3$ . Due to the importance of the material composition of the guide tubes, the data from the mill specifications are provided in Table 2.1. The outside diameter of the tubes is  $0.6462 \pm 0.0028 \text{ cm}$  and the inside diameter is  $0.6083 \pm 0.0013 \text{ cm}$ .

The control and safety blades were external to the vessel and were withdrawn during the neutron flux measurements taken in the course of the critical approach. The experiments with the boiler tube-type tank were conducted with the reflector tank containing water. The reflector tank was filled to a level 56 cm below the top of the boiler tube-type tank. The distance between the boiler tube-type tank bottom (outside surface) and the bottom of the reflector tank (inside surface) is  $16.0 \pm 0.2 \text{ cm}$ .

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

Engineering drawings for the boiler tube-type tank assembly are provided in Appendix B.

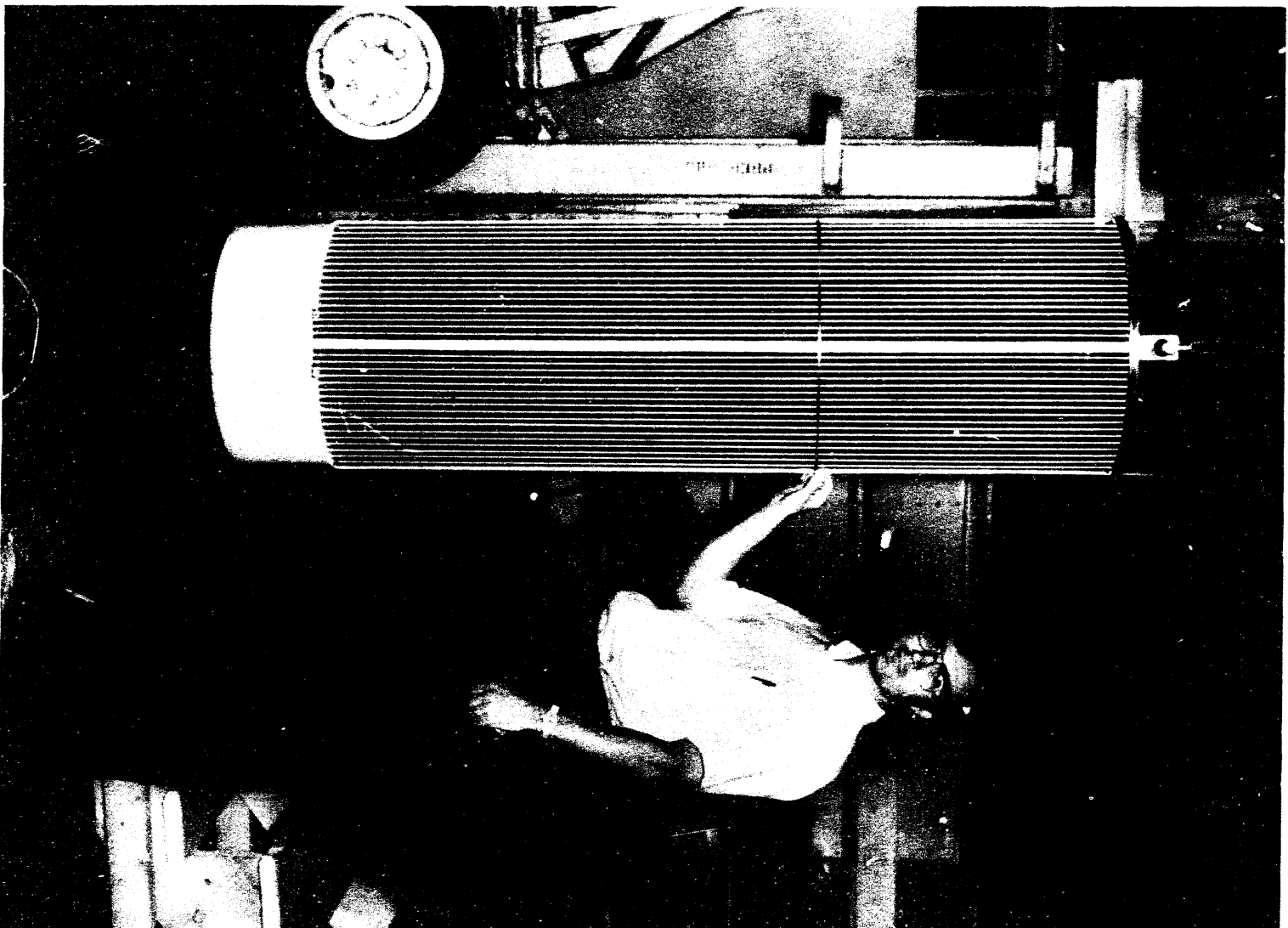


FIGURE 2.10 Photograph of Completed Boiler Tube-Type Inner Assembly



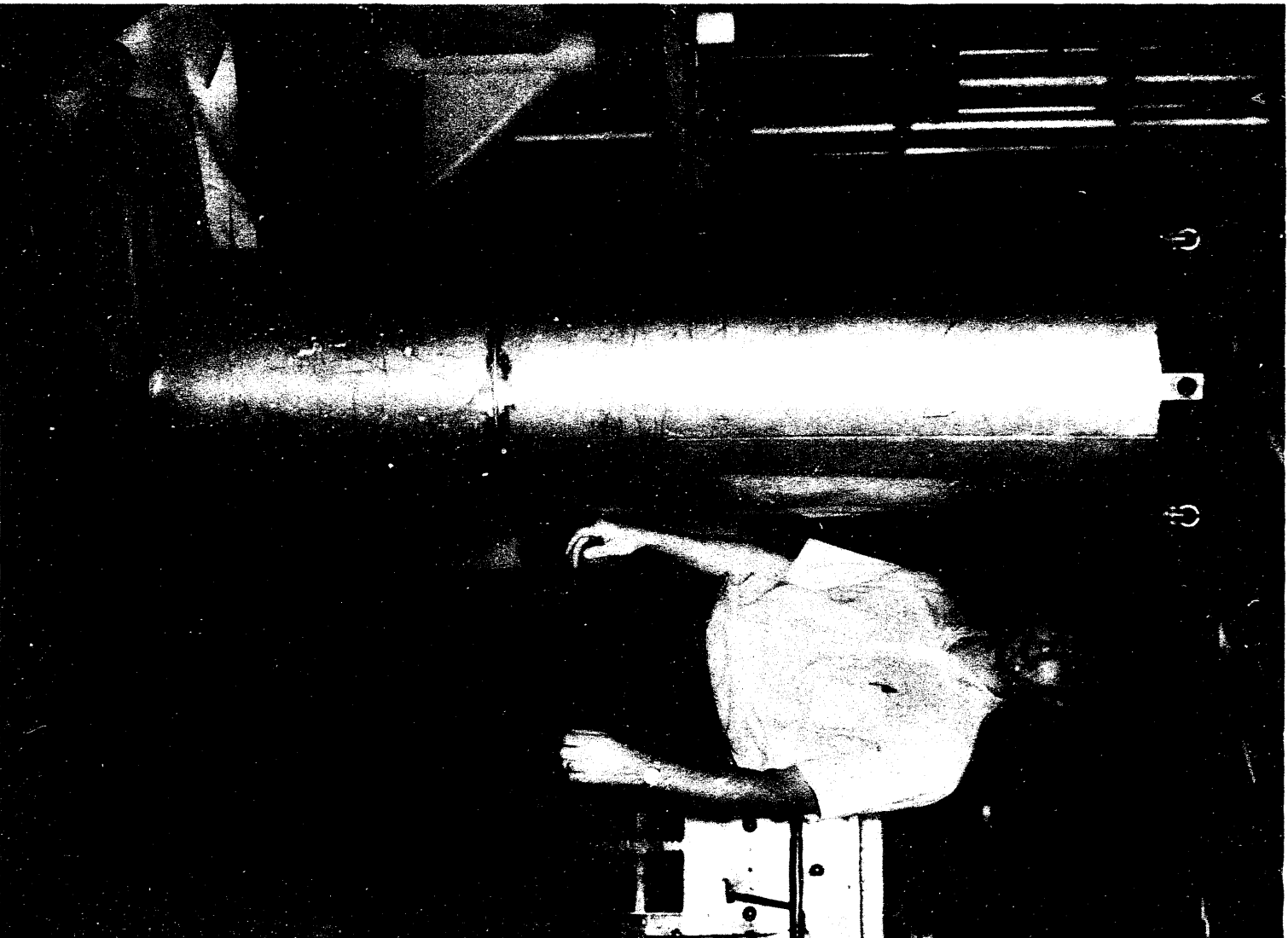


FIGURE 2.11 Photograph of Completed Boiler Tube-Type Tank

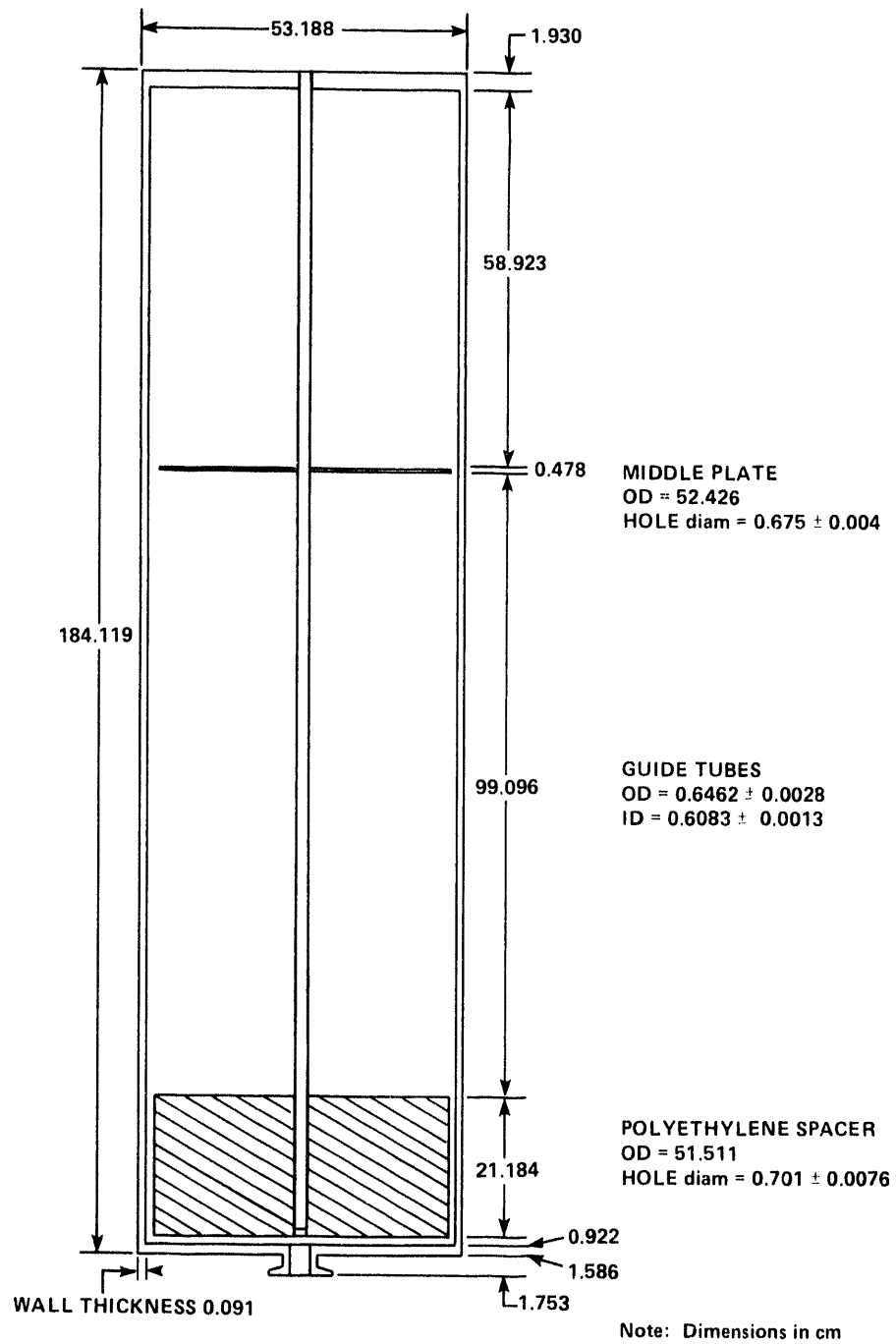


FIGURE 2.12 Schematic of Boiler Tube-Type Tank Showing As-built Dimensions

TABLE 2.1 Mill Specification Data for Type 304L Stainless Steel Guide Tubes

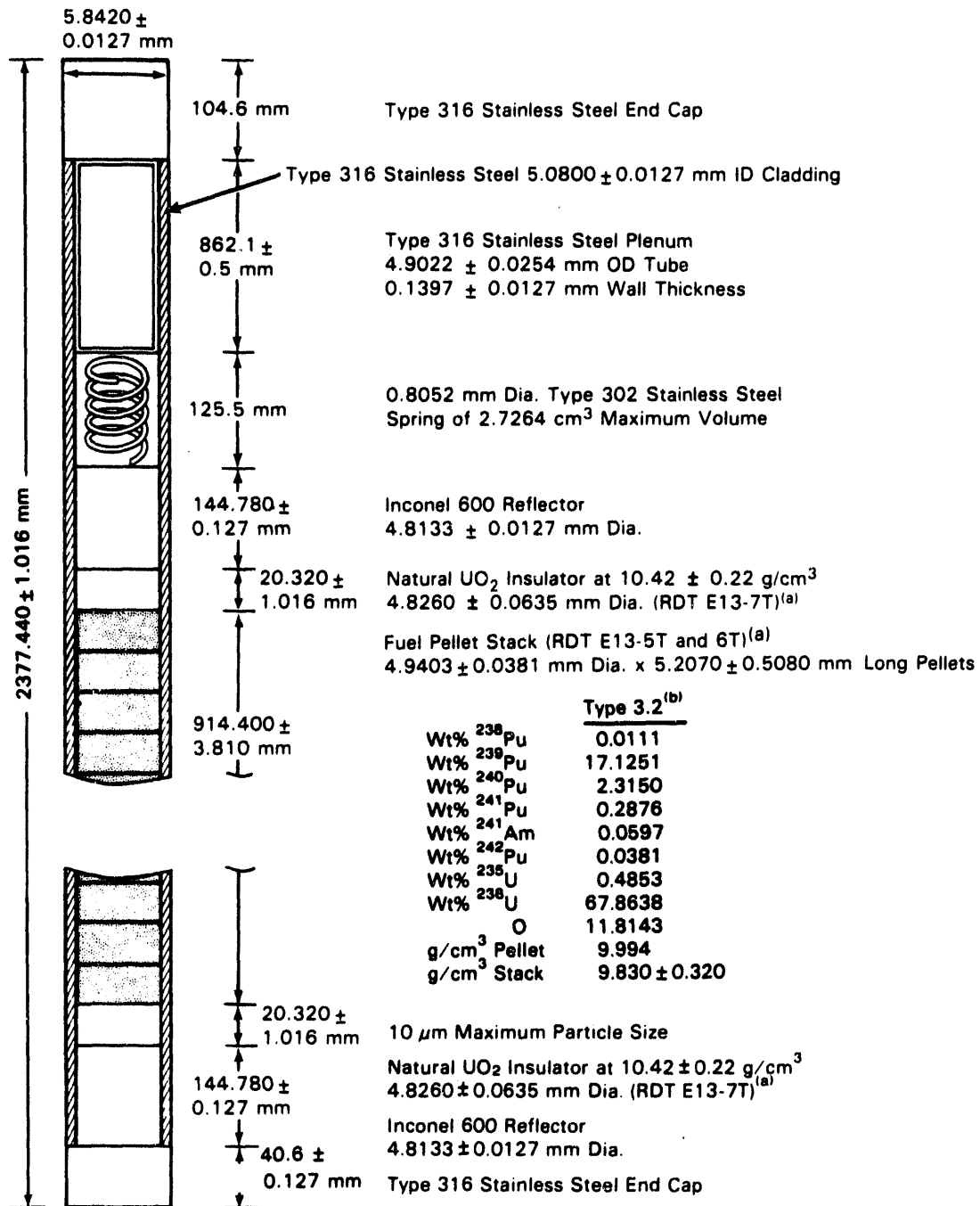
<u>Element</u>	<u>Wt%</u>
Fe	69.208 <sup>(a)</sup>
Cr	18.33
Ni	9.33
Mn	1.54
Si	0.47
Mo	0.31
Cu	0.29
C	0.24
Co	0.19
N	0.060
P	0.027
S	0.005

---

(a) Balance of data to equal 100 wt%.

### 2.3 FFTF FUEL PINS

The critical experiments were conducted with Type 3.2 FFTF mixed oxide fuel pins. These fuel pins were used in other critical experiments that have been performed at the PNL-CML (Bierman 1986, Bierman 1979, and Durst 1980). The fuel pins were referred to as Fast Test Reactor (FTR) pins in these reports. The Type 3.2 FFTF fuel pins contain 19.78 wt% plutonium. The isotopic and density data for these particular fuel pins are given in Figure 2.13. These data are based on measured data from pellet lots used in fabricating the fuel and are more accurate than the information previously reported (Bierman 1979, Durst 1980, and Primm 1980). The dimensions of the fuel pins are also given in Figure 2.13. The actual MOX fuel region is restricted to a 91.4 cm length near the bottom of the pins. The remainder of the pin consists of Inconel reflectors, end caps and other hardware. As discussed in the previous section, the elevation of the MOX fuel should be considered equal to the top of the polyethylene spacer of the boiler tube-type tank.



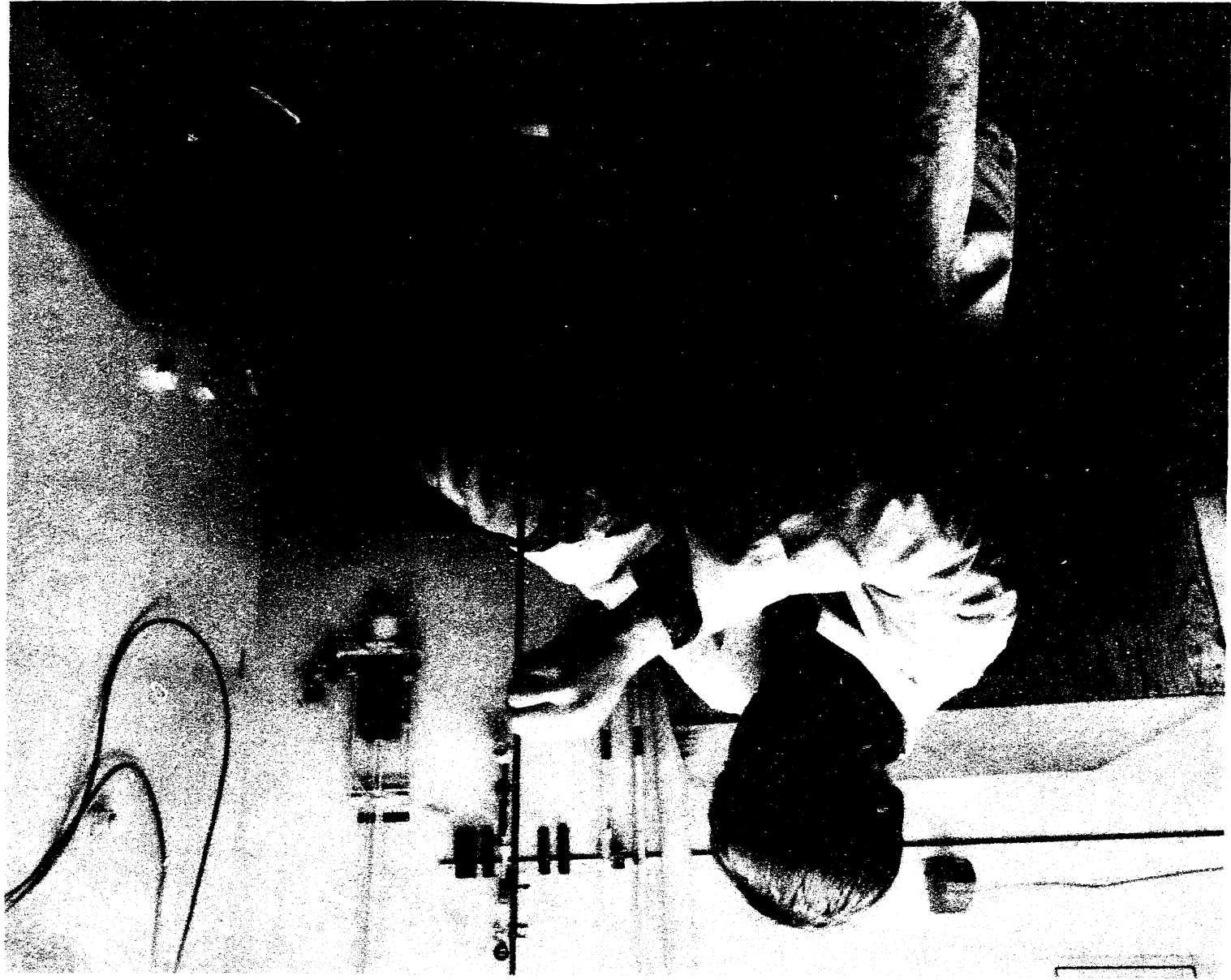
<sup>(a)</sup> Referenced Division of Reactor Development and Technology standard.

<sup>(b)</sup> Isotopics based on measured data from pellet lots. Americium-241 contents as of January 1, 1976 based on Plutonium-241 half life of 14.35 yrs.

FIGURE 2.13 Simplified Description of Type 3.2 FFTF Fuel Pin

Figure 2.14 shows the loading of a fuel pin into the boiler tube-type tank prior to mounting on the experimental assembly. Figure 2.15 shows the boiler tube-type assembly loaded with 996 fuel pins.

FIGURE 2.14 Photograph of the Boiler Tube-Type Tank Showing Insertion of a Mixed Oxide Fuel Pin



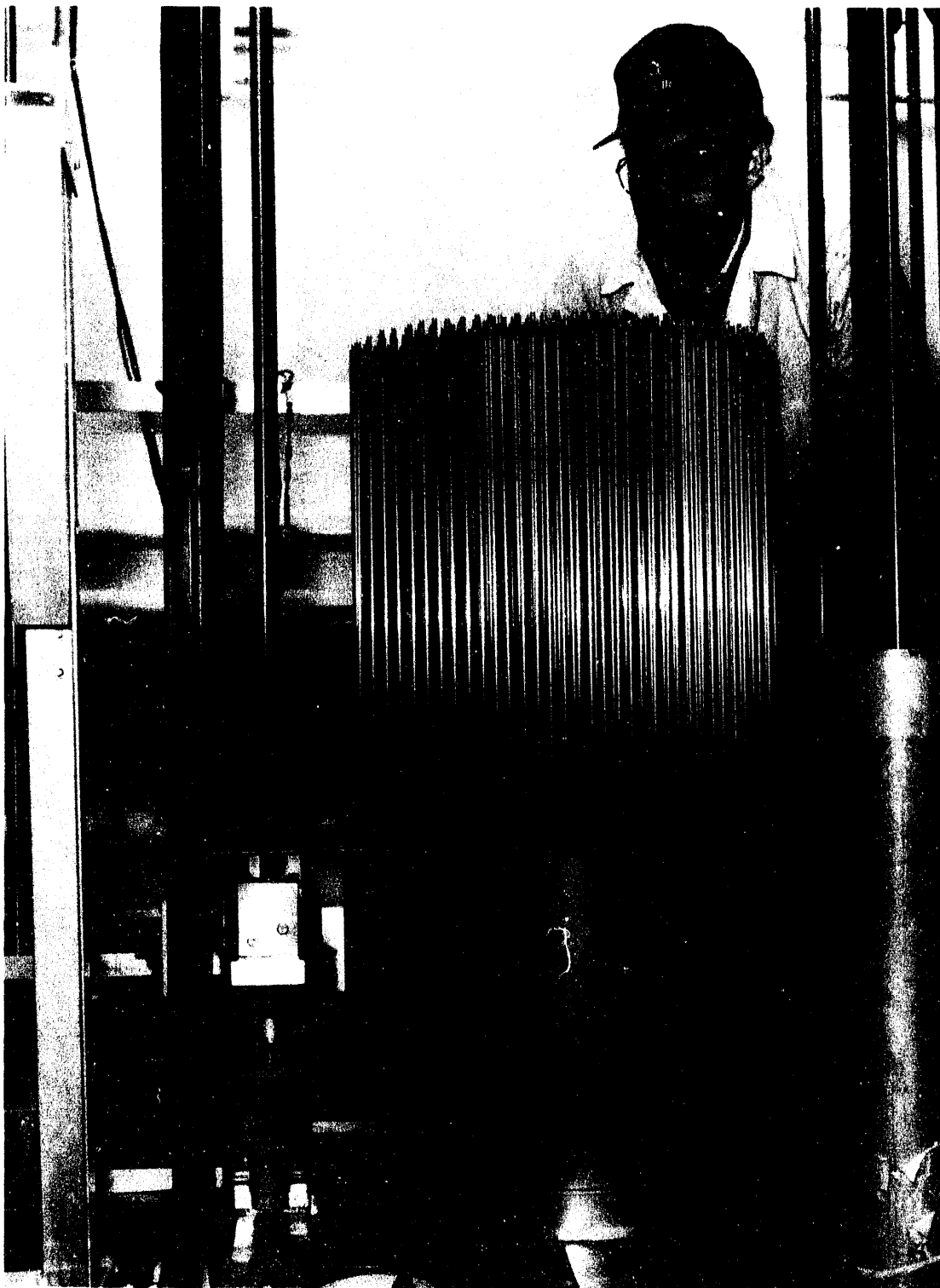


FIGURE 2.15 Photograph of the Boiler Tube-Type Tank Loaded with Mixed Oxide Fuel Pins

### 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 TECHNIQUE

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. At delayed critical condition the neutron count rate approaches infinity so that 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 solution height at near critical values. The computer calculated least square fits of the inverse multiplication curves, used in determining the critical value for the solution height in each experimental assembly, are included in Appendix C.

#### 3.2 CRITICALITY DATA

The criticality data for this report were obtained between November 1987 and April 1988, when thirteen experiments were completed using the boiler tube-type tank system. The data are summarized in Table 3.1. Figure 3.1 shows a plot of the critical heights determined at various concentrations of the plutonium-uranium nitrate over a range of ~4 to 468 g (Pu + U)/liter in the boiler tube-type assembly loaded with the mixed oxide fuel pins. Figure 3.2 shows a plot of the critical heights determined for a concentration of ~464 g (Pu + U)/liter for various additions of gadolinium from ~0 to 2.2 g Gd/liter. The sample analysis methods and descriptive titles are given in Table 3.2. The critical heights were calculated by a least squares fit to the inverse neutron multiplication data from three neutron detectors (computer printout provided in



**TABLE 3.1** Criticality Measurements with Plutonium-Uranium Nitrate Solution in the Boiler Tube-Type Tank<sup>(a)</sup>

<u>Run Date</u>	<u>Project Case Number</u>	<u>CML Experiment Number</u>	<u>Sample Number</u>	<u>Pu (g/liter)</u>	<u>U (g/liter)</u>	<u>Gd (g/liter)</u>	<u>Density<sup>(b)</sup> (g/cm<sup>3</sup>)</u>	<u>Free Acid (M)</u>	<u>Critical Height<sup>(c)</sup> (cm)</u>
11/04/87	51C	106	1230	0.88	2.7	0.0	1.0170	0.41	18.41
11/05/87	51CR	106R	1230	0.88	2.7	0.0	1.0170	0.41	18.55
11/09/87	41	107	1231	73.95	254.09	0.0	1.4833	0.57	21.34
11/19/87	51	109	1233	47.50	163.06	0.0	1.3168	0.52	20.01
11/24/87	52	110	1234	22.03	77.84	0.0	1.1577	0.47	18.90
11/25/87	52R	110R	1234	22.03	77.84	0.0	1.1577	0.47	19.03
12/02/87	40	111	1235	103.70	303.98	0.0	1.6784	0.66	23.07
12/09/87	65	112	1236	103.19	300.62	0.49	1.6758	0.65	30.28
12/11/87	66	113	1237	102.23	359.69	0.98	1.6748	0.65	38.78
12/17/87	70	114	1238	102.65	350.59	1.47	1.6745	0.66	51.06
12/23/87	70C1	115	1239	102.68	359.55	1.97	1.6748	0.65	73.08
12/31/87	70C2	116	1248	103.61	362.45	2.16	1.6790	0.65	90.27
04/04/88	---	117	1263	83.30	286.57	0.0	1.5507	0.90	27.42

(a) Boiler tube-type tank contained 996 FFTF Type 3.2 pins except for experiment 117 where the fuel pins were removed. The vessel was water reflected.

(b) Density measured at 23°C

(c) Zero reference is the top of the polyethylene spacer.

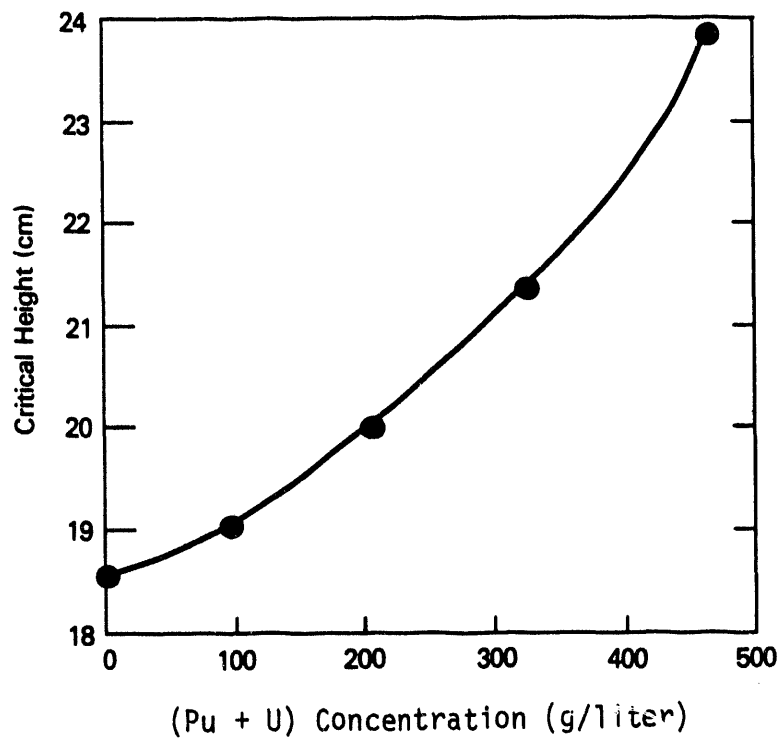


FIGURE 3.1 Effect of Plutonium and Uranium Concentration on Critical Height

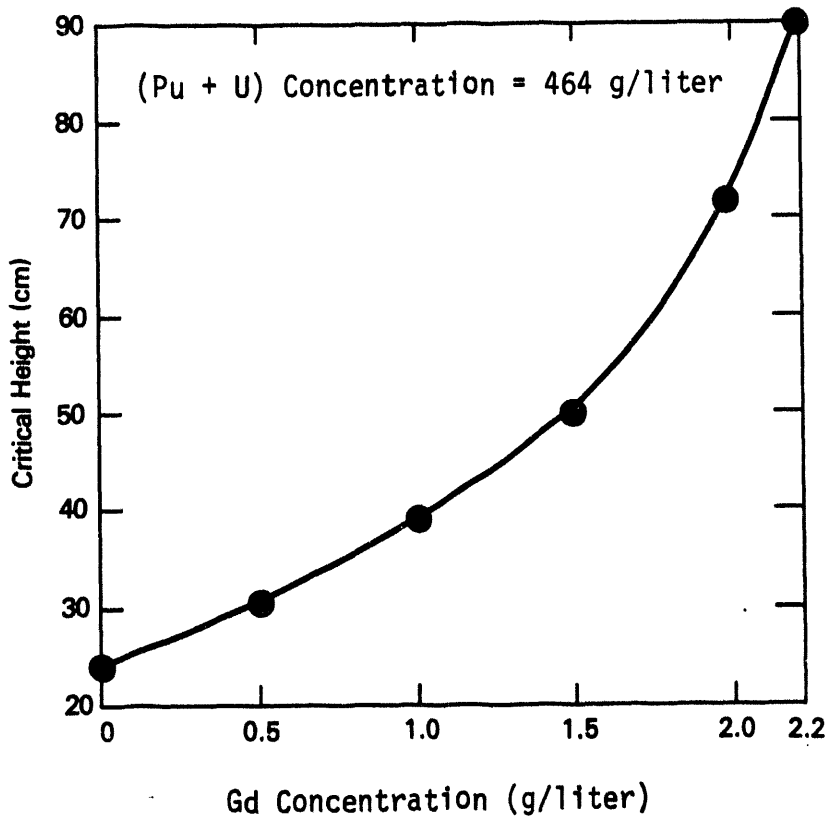


FIGURE 3.2 Effect of Gadolinium Concentration on Critical Height

TABLE 3.2 Chemical Analysis Methods

<u>Measurement</u>	<u>Method Title<sup>(a)</sup></u>	<u>Date of Approval</u>
Plutonium	Plutonium by Automated Amperometric Titration. (38.3)	03/18/85
Plutonium	Plutonium Purification by Ion Exchange. (38.9)	03/11/86
Uranium	Uranium by Automated Potentiometric Titration. (38.8)	02/05/86
<sup>241</sup> Am	Americium-241 by Anion Exchange and Alpha Analysis. (48.18)	05/14/75
Free Acid	Determination of Free Acid in Uranium/Plutonium Solutions. (Using an improved oxalate method) (48.22)	02/04/86
Density	Density of Solutions. (Using Mettler/ Paar Density Meter) (48.23)	02/05/86
Isotopic	Isotopic Composition of Plutonium and Uranium by Mass Spectroscopy. (38.6)	09/27/78
Gd, Impurities	ICP Analysis. <sup>(b)</sup> (SP-78)	04/01/86
Gd, Gd Hydrate	ICP Analysis. <sup>(c)</sup> (HTA-4-1)	07/01/87
Gd Hydrate	Gravimetric. (PNL MA-597 Vol. 2)	05/30/75

(a) The numbers in brackets are method numbers for PNL MA-597, Vol 2, except 48.22 from Vol. 7.

(b) This method was used to determine gadolinium in experiment solution.

(c) This method was used to determine gadolinium in the concentrated gadolinium nitrate solution.

Appendix C). The  $^{241}\text{Am}$  content for each sample analyzed, the analysis date and the experiments covered by that sample are given in Table 3.3. The isotopic analysis values for the plutonium-uranium nitrate solutions used in the experiments are given in Table 3.4. Table 3.5 provides information on the temperatures of the critical assembly room (CAR), the dump mix tank (DM) and the water reflector. Also in Table 3.5, 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 chemical analysis data and analysis calculations for the gadolinium in the experimental solutions.

Appendix E provides data on the chemical analyses for the impurities found in the plutonium-uranium nitrate solutions.

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

### 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 computer code 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 (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.

TABLE 3.3 Chemical Analysis Values for Americium-241

<u>Sample Number</u> (a)	<u><sup>241</sup>Am (ug/ml)</u>	<u>Analysis Date</u>
1232	265	12/16/87
1235	573	12/16/87
1263	453	04/29/88

---

(a) The analysis results from samples 1232, 1235 and 1263 can be used to estimate the <sup>241</sup>Am concentrations for experiments 106, 106R, 107, 110 and 110R using the ratio based on plutonium concentration. The plutonium concentration for sample 1232 was 47.08 g Pu/liter.

Sample 1232 covers experiment 109

Sample 1235 covers experiments 111 to 116

Sample 1263 covers experiment 117

**TABLE 3.4** Isotopic Analysis Values of Plutonium and Uranium<sup>(a)</sup>

	<u>Sample 1232</u>	<u>Sample 1235</u>
<u>Pu</u> <sup>(b)</sup>	12/14/87	12/14/87
238	0.046 ± 0.005	0.032 ± 0.004
239	91.11 ± 0.04	91.12 ± 0.04
240	8.33 ± 0.04	8.33 ± 0.04
241	0.419 ± 0.02	0.419 ± 0.02
242	0.095 ± 0.001	0.096 ± 0.001
<u>U</u> <sup>(c)</sup>	12/14/87	12/14/87
238	99.234 ± 0.008	99.232 ± 0.008
236	0.053 ± 0.005	0.053 ± 0.005
235	0.705 ± 0.008	0.709 ± 0.008
234	0.008 ± 0.001	0.007 ± 0.001

(a) All values given in wt%. An average of these isotopic results should be used for all experiments.

(b) Date of Pu analysis

(c) Date of U analysis

TABLE 3.5 Information on Temperature, Reflector Level, and Control and Safety Blade Position

<u>Experiment Number</u>	<u>Temperature °C</u>			<u>Reflector Level Distance Below Vessel Top (cm)</u>	<u>Control and Safety Blade Distance Below Vessel Top (cm)</u>
	<u>CAR</u>	<u>DM Tank</u>	<u>Reflector</u>		
106	25.2	24.1	24.0	56	47
106R	24.2	24.0	23.7	56	47
107	24.3	23.9	23.3	56	47
109	24.0	22.6	21.4	56	47
110	24.1	22.4	21.3	56	47
110R	23.6	22.5	22.1	56	47
111	23.6	24.4	20.6	56	47
112	23.9	24.7	19.1	56	47
113	23.6	24.7	21.9	56	47
114	22.7	24.2	17.9	56	47
115	22.6	23.4	17.4	56	47
116	22.9	23.0	18.1	56	47
117	26.2	22.3	21.4	56	47



The latest estimated values of uncertainties are listed in Table 3.6.

**TABLE 3.6 Estimate of Measurement Uncertainties**

Pu Concentration	± 0.2%
U Concentration	± 0.2%
Density	± 0.0003 g/cm <sup>3</sup>
Free Acid	± 0.04 M
Critical Height	± 1.6 mm

The uncertainty values for the chemical analyses were provided by M. C. Burt of the Chemical and Analysis Section. The critical height uncertainty is given as 1.6 mm although the least square fitting of approach data for three counting systems would indicate a smaller value as reasonable. The 1.6 mm unit is the smallest 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.

- 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 and M. A. Covert for providing valuable advice, and assistance in performing the experiments.
- D. A. Costanzo for providing advice regarding chemical analysis methods.
- C. A. Strand for providing as-built data on the FFTF fuel pins.
- L. N. Terry for typing, proofreading and guidance in preparation of this report.

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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 H<sub>2</sub>O 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

ENGINEERING DRAWINGS OF THE BOILER TUBE-TYPE TANK ASSEMBLY

APPENDIX C

LEAST SQUARE FITS OF THE CRITICAL APPROACH DATA

## APPENDIX C

### LEAST SQUARE FITS OF THE CRITICAL APPROACH DATA

The extrapolated values given are for solution height in the boiler tube-type tank. The solution height values used in these plots are given in inches. The solution height is the height of the solution above the polyethylene spacer which is also the level where the mixed oxide fuel in the fuel pins begin.



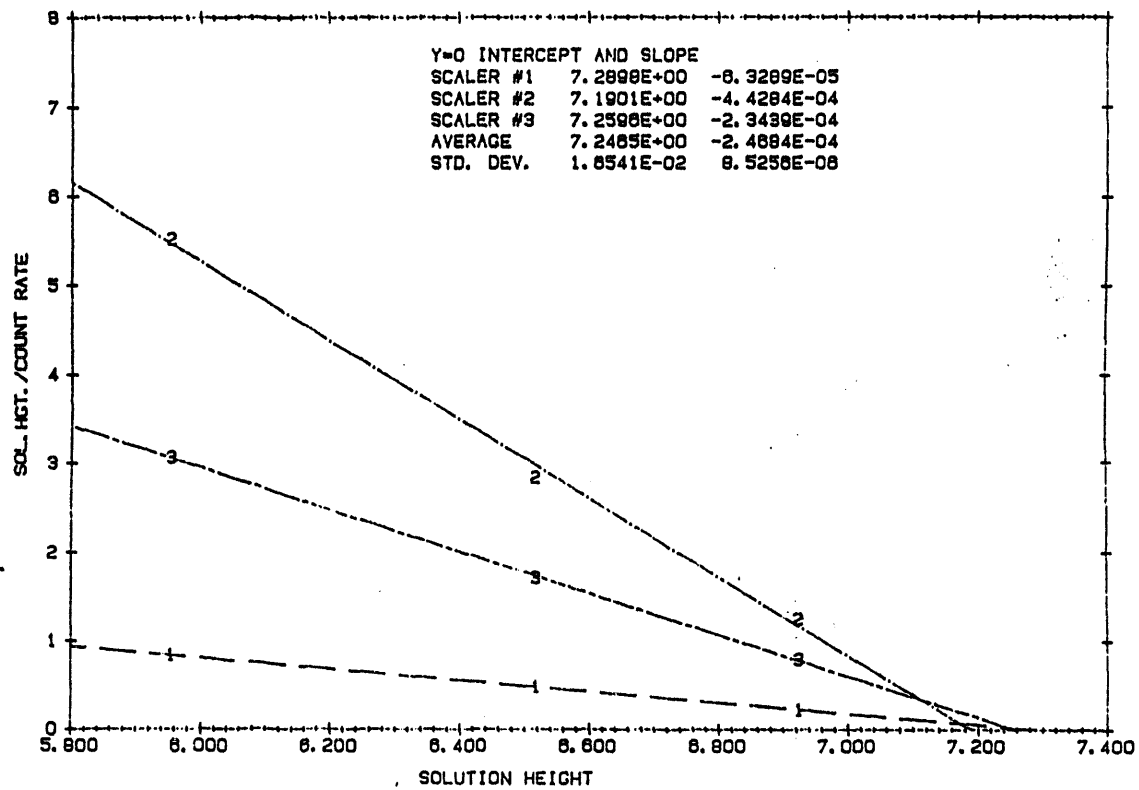


FIGURE C.1 Least Square Fits for CFRP-PNC 106

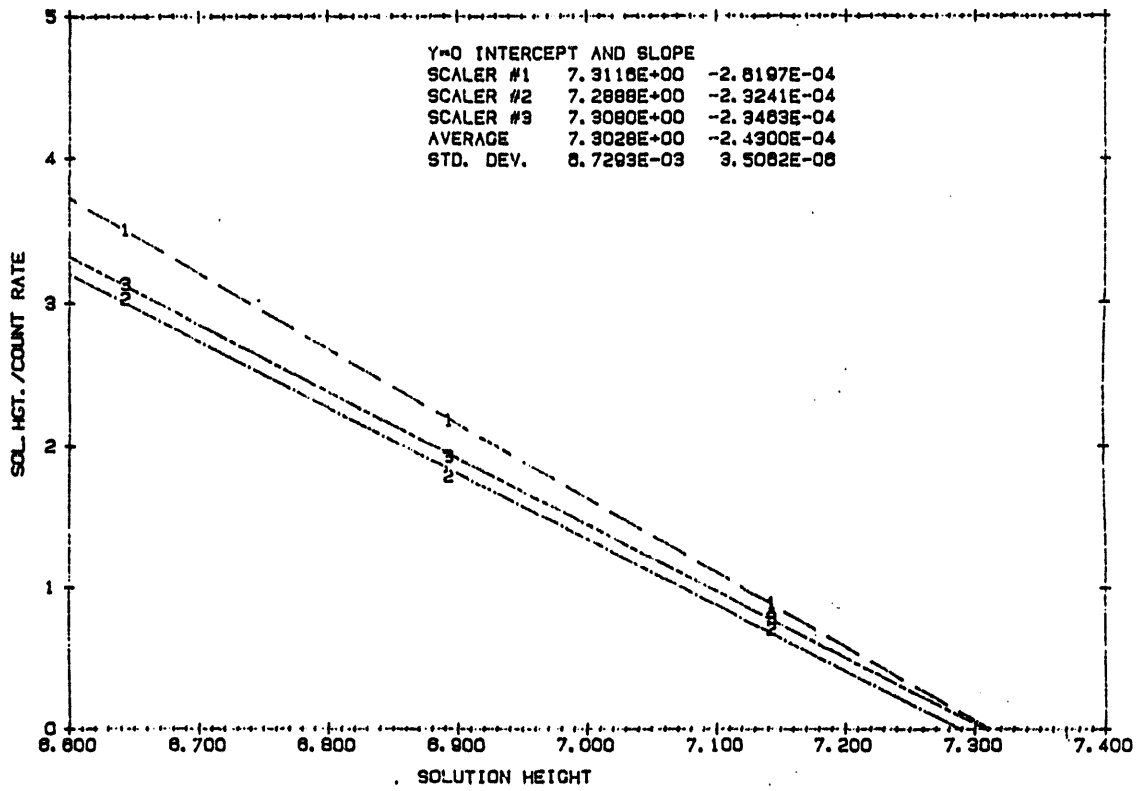


FIGURE C.2 Least Square Fits for CFRP-PNC 106R

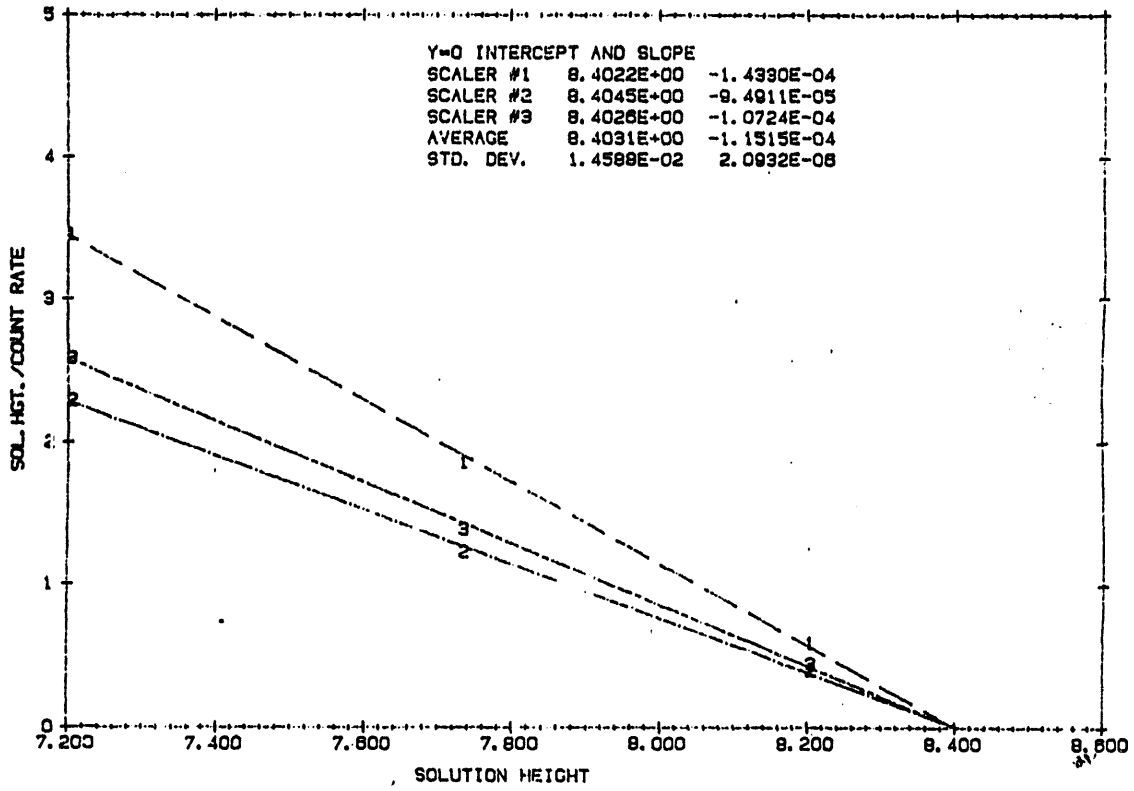


FIGURE C.3 Least Square Fits for CFRP-PNC 107

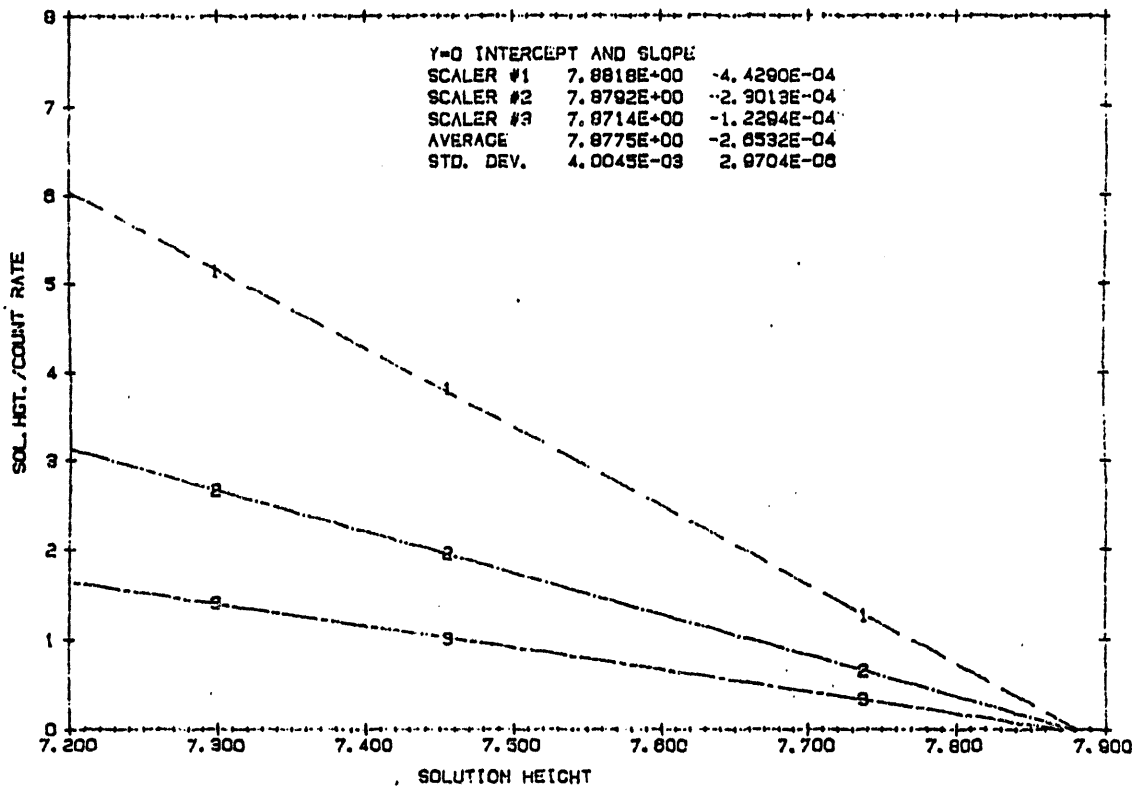


FIGURE C.4 Least Square Fits for CFRP-PNC 109

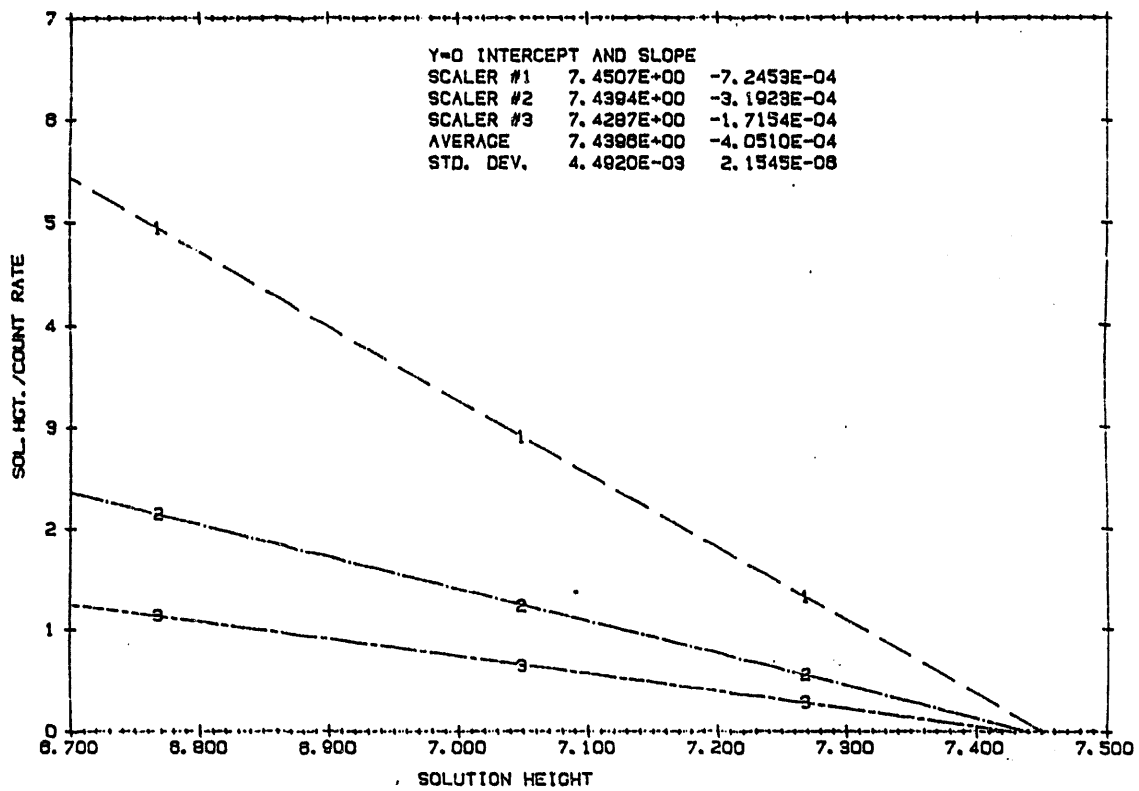


FIGURE C.5 Least Square Fits for CFRP-PNC 110

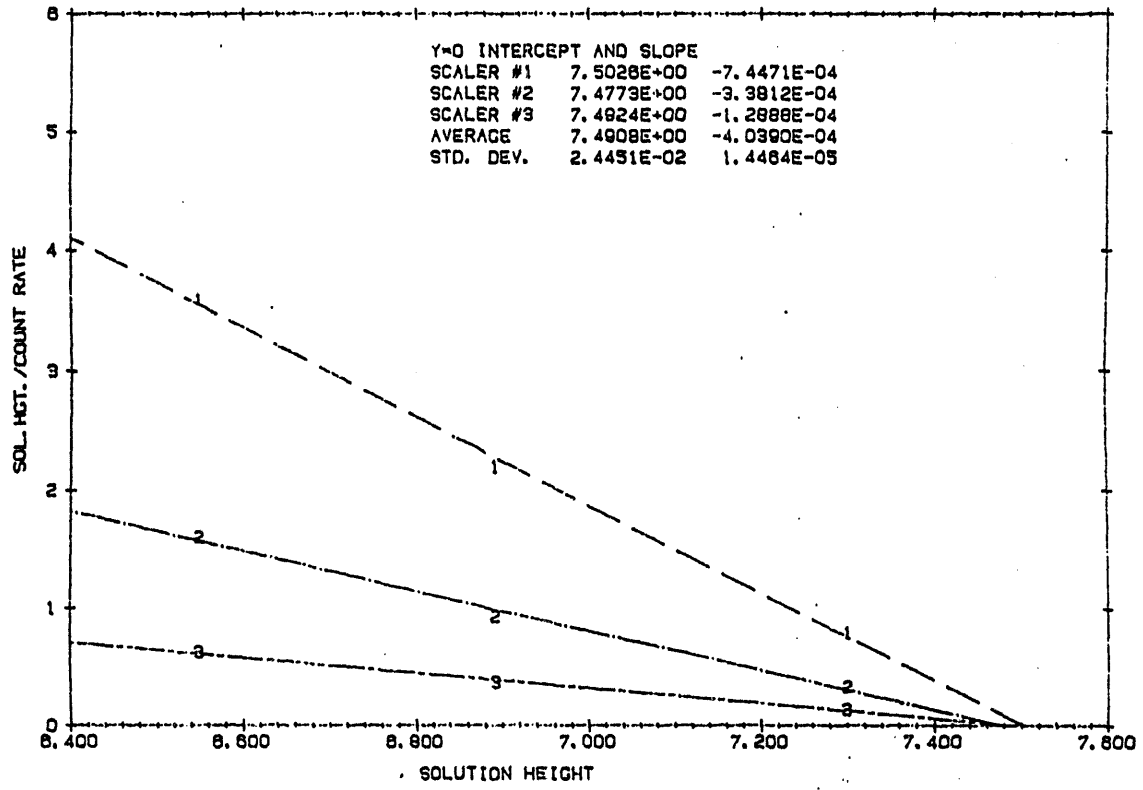


FIGURE C.6 Least Square Fits for CFRP-PNC 110R

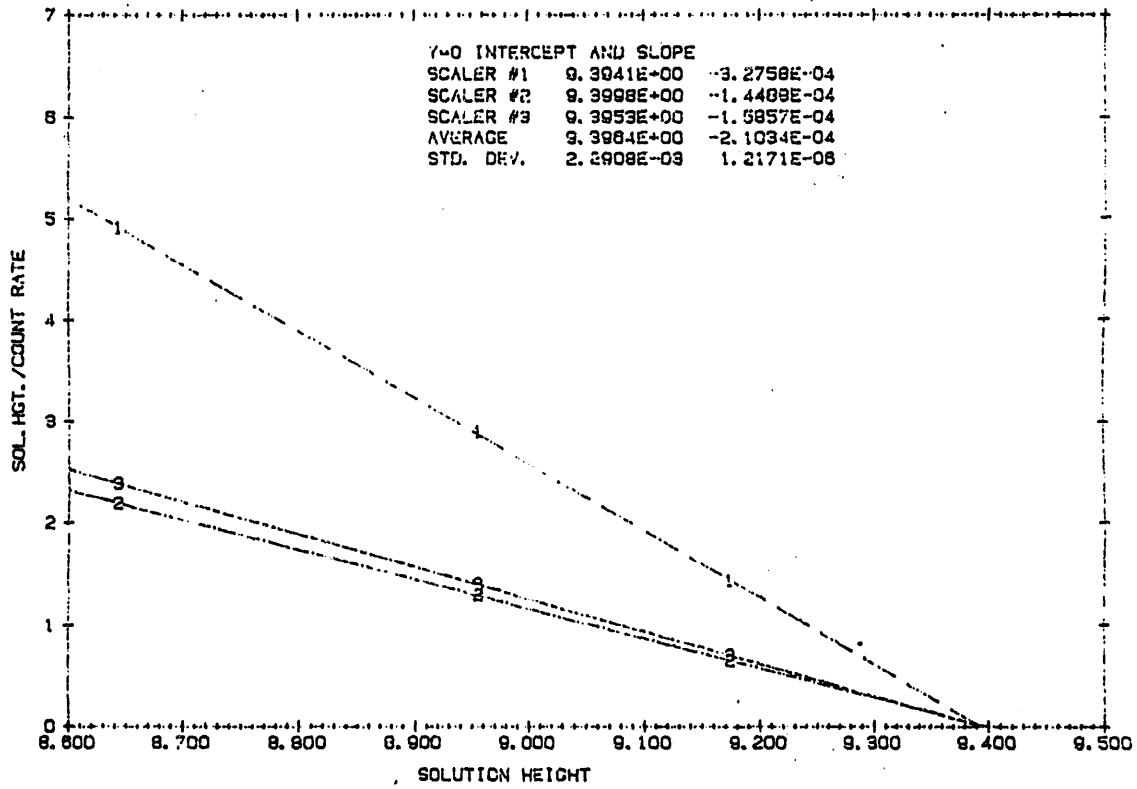


FIGURE C.7 Least Square Fits for CFRP-PNC 111

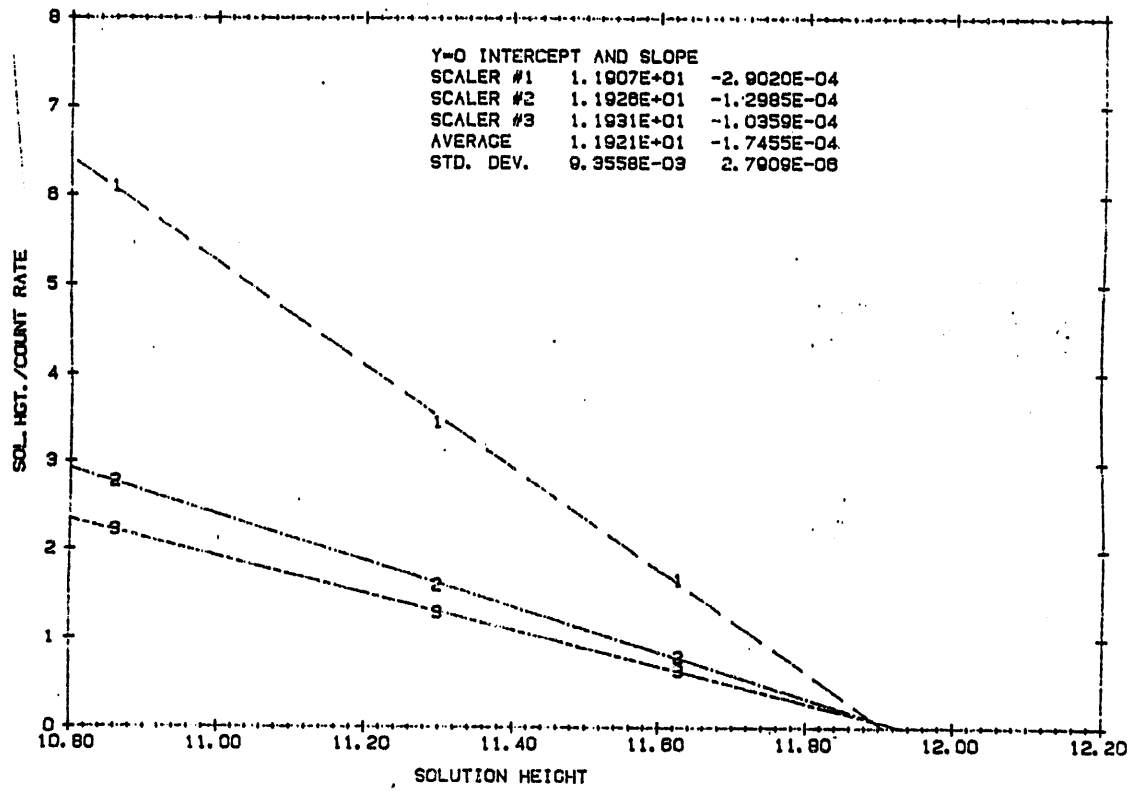


FIGURE C.8 Least Square Fits for CFRP-PNC 112



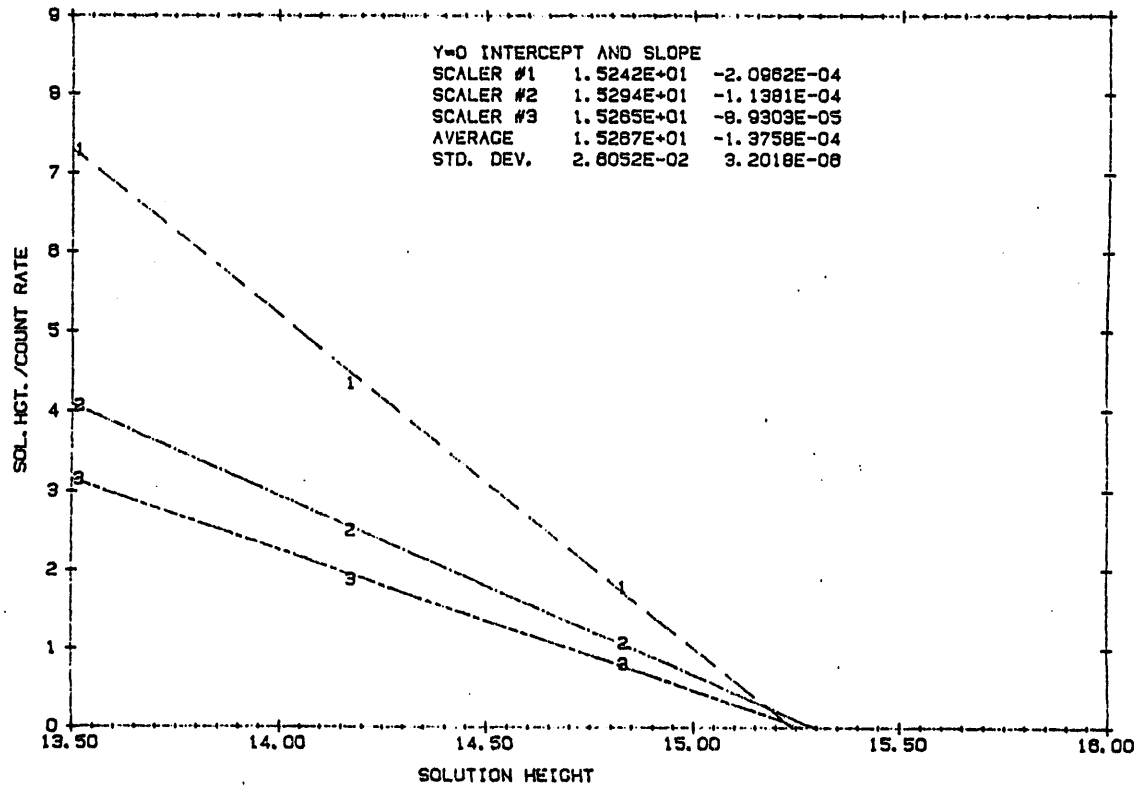


FIGURE C.9 Least Square Fits for CFRP-PNC 113

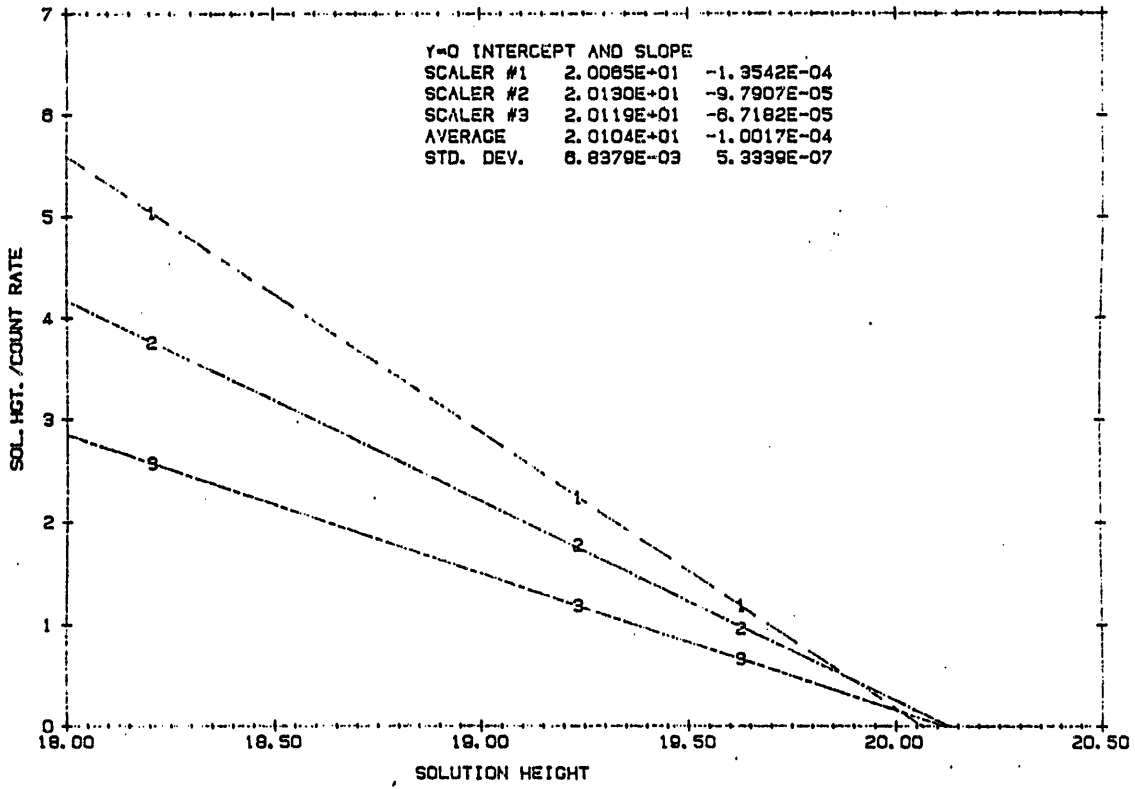


FIGURE C.10 Least Square Fits for CFRP-PNC 114

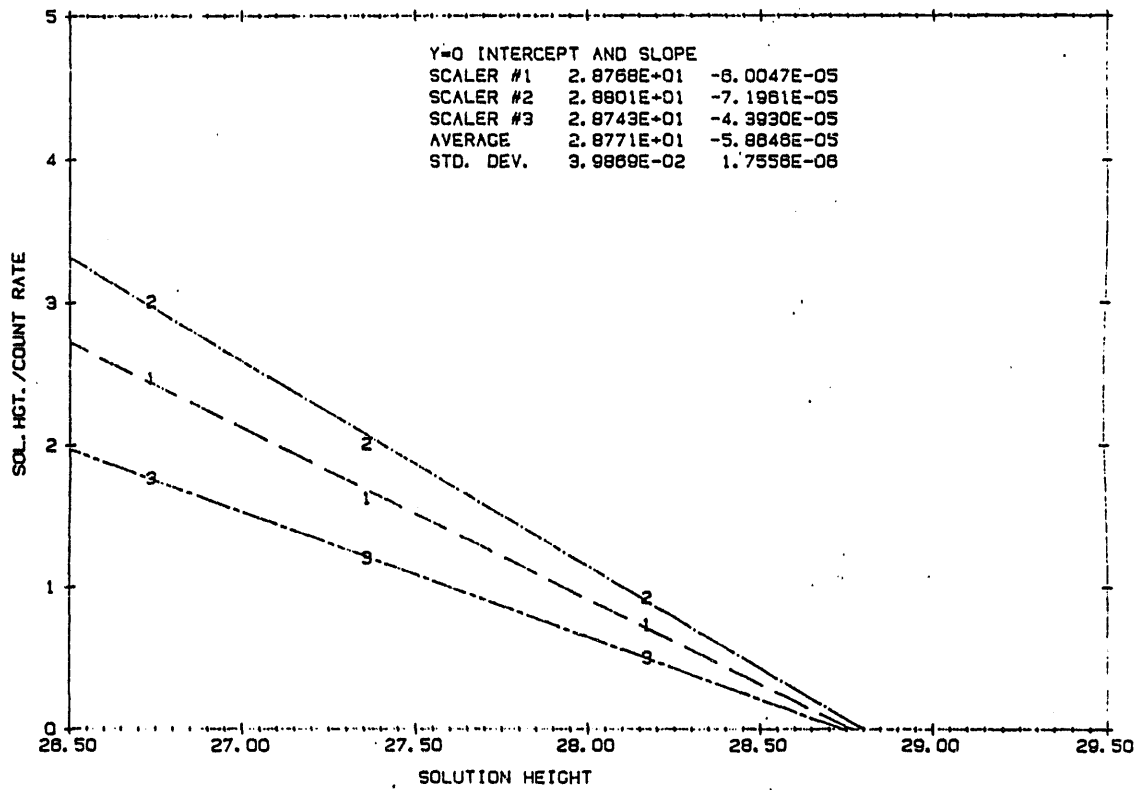


FIGURE C.11 Least Square Fits for CFRP-PNC 115

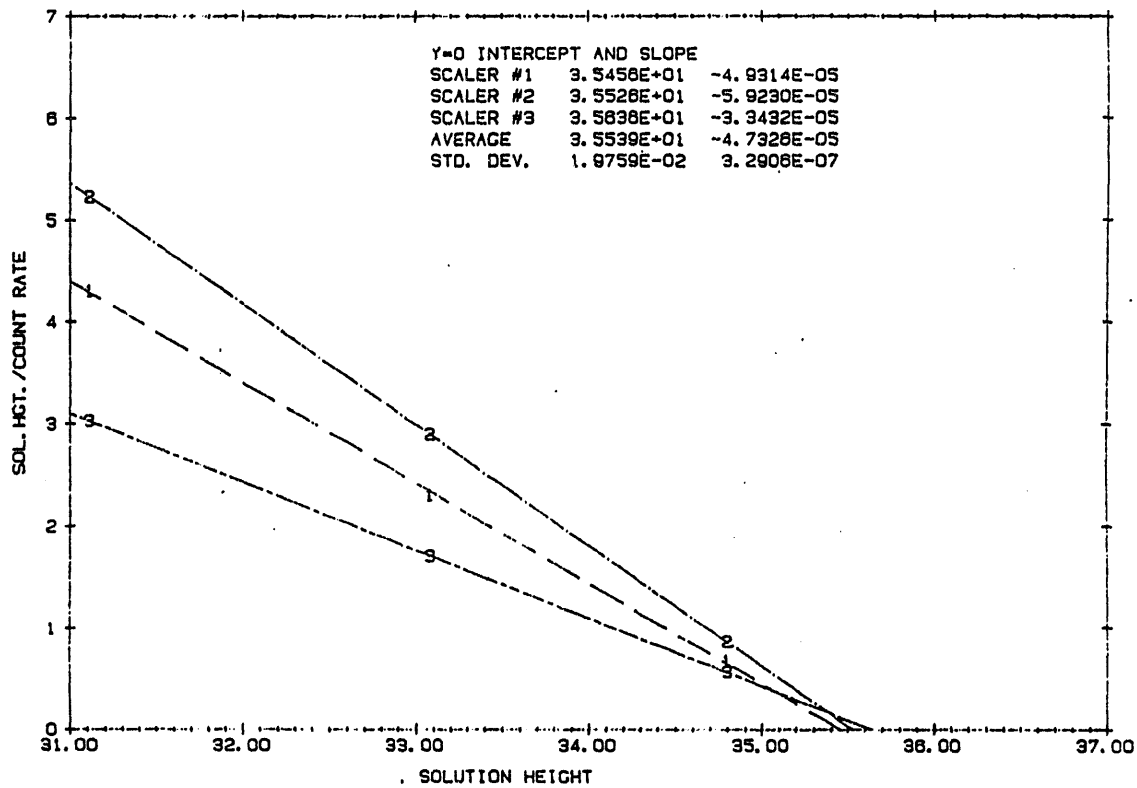
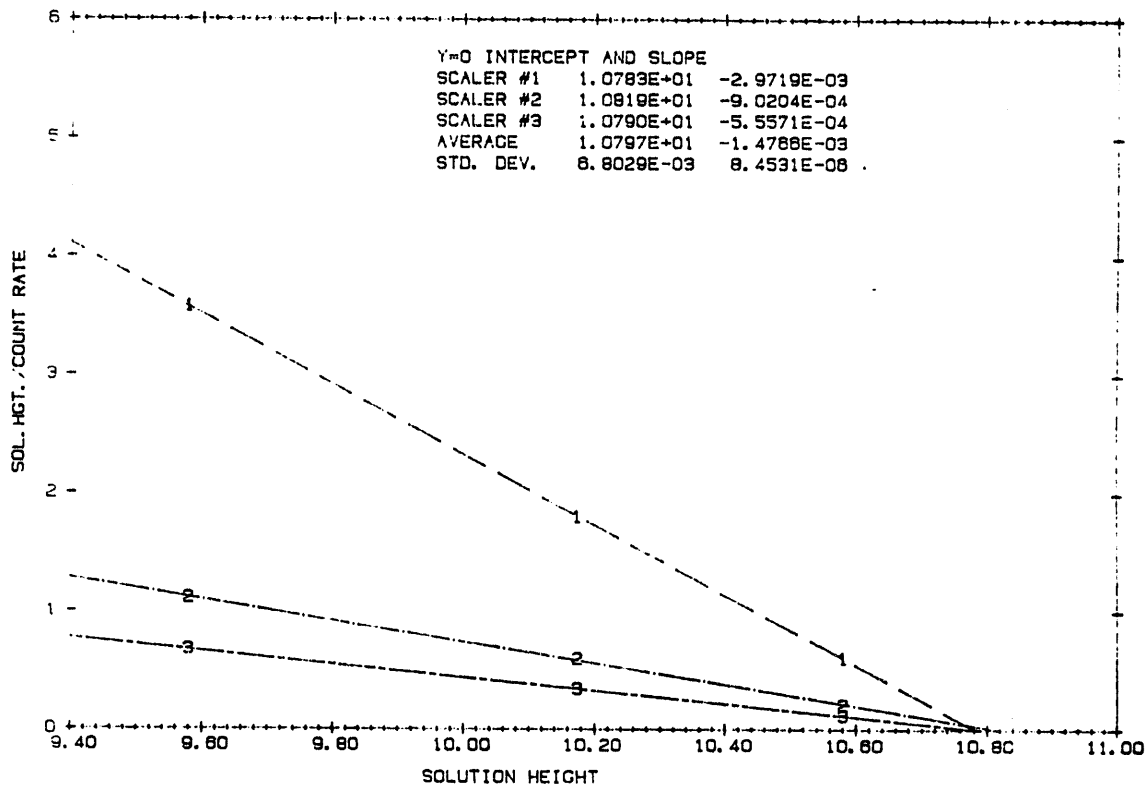


FIGURE C.12 Least Square Fits for CFRP-PNC 116



**FIGURE C.13** Least Square Fits for CFRP-PNC 117

APPENDIX D

ANALYSIS OF GADOLINIUM CONCENTRATION DATA

## APPENDIX D

### ANALYSIS OF GADOLINIUM CONCENTRATION DATA

Initially, it was necessary to determine the amount of gadolinium to add to the fissile solution so that the experiment solutions would have gadolinium concentrations close to expected values. The gadolinium was added in the form of gadolinium nitrate powder,  $(\text{Gd}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O})$ , so it was necessary to evaluate the fraction of gadolinium in the molecule. Based on two independent analyses, Loss on Ignition and Inductively Coupled Plasma Spectroscopy (ICP), the fraction of gadolinium to gadolinium nitrate powder was determined to be  $0.367 \pm 0.006$ , and the amount of water,  $x$ , was estimated to be  $4.8 \pm 0.4$ . Using measured amounts of gadolinium nitrate powder, water and nitric acid, five concentrated gadolinium solutions were prepared. The quantities of materials used to prepare the concentrated gadolinium solutions are given in Table D.1. This table also gives the amount of gadolinium and volume of concentrated solution that was added to the bulk fissile solution stored in the dump mix tank to obtain the diluted gadolinium concentrations used in the critical experiments. For example, to obtain the 0.5 g Gd/liter solution, 1.1029 liters of concentrated solution with 107.69 g of gadolinium was added to the existing volume (214 liters) of fissile solution in the dump mix tank. This process was continued with each of the concentrated gadolinium solutions to obtain the progressively higher gadolinium concentrations used in the critical experiments. As a check, estimates of the diluted gadolinium concentrations for solutions used in the critical experiments were made and are given in Table D.1. Although the precision of these results is very good, the accuracy of the gadolinium concentration is approximately  $\pm 0.2\%$  due to the uncertainty in the amount of gadolinium associated with the gadolinium nitrate powder.

A second method of determining the gadolinium concentrations of the experiment solutions used analytical data from the concentrated gadolinium solutions that were prepared. The densities and gadolinium concentrations of

these solutions were measured and are given in Table D.2. Since these solutions did not contain plutonium and uranium the ICP analyses were simplified. Estimates of the gadolinium concentrations of the experimental solutions were made using the analytical data given in Table D.1 (column 7, volume) and D.2 (gadolinium concentration). The gadolinium concentration estimates are given in Table D.3. The uncertainty in these results is estimated to be  $\pm 2\%$ .

A third method of determining the gadolinium concentrations was based on an ICP analysis of samples of the experimental solutions. However, the analysis of gadolinium concentration in the critical experiment solutions was difficult because the plutonium and uranium had to be separated from the solution prior to assay. The separation was performed by contacting the sample solutions with an equal volume of 30% tributyl phosphate in hexane. The organic layer was discarded after each phase separation. The solution was adjusted to 3M acid and two contacts were made. Two more contacts were made after an acid adjustment to 4M. The extraction process also removes some of the gadolinium from the aqueous solution so that analytical studies were performed to evaluate the characteristics of the recovery curve over the concentrations of interest (i.e., 0-2 g/liter). The results of the studies showed that 10 - 20% of the gadolinium was lost in the separation procedure. The large variation in the amount of gadolinium that was recovered for each sample resulted in a high degree of uncertainty in the results obtained with this procedure. To determine the recovery (calibration) curve for the ICP analysis of the critical experiment solutions, 1 ml quantities of sample number 1235 were spiked with 0, 500, 1000, 1500, and 2000  $\mu\text{g}$  Gd. The results of this analysis are shown in Table D.4. The following equation, based on a linear regression analysis of the data, was used to determine the actual gadolinium concentrations from the ICP results:

$$x = 1.1709y + 0.01614$$

where  $y$  is the concentration of gadolinium in g/liter as determined by the ICP analysis, and  $x$  is the actual gadolinium concentration in g/liter. The coefficient of determination,  $r^2$  is 0.996. The results from the ICP analysis



of the critical experiment solutions and the computed actual gadolinium concentrations are given in Table D.5. The uncertainty in these results is estimated to be  $\pm 4\%$ .

A summary of the estimated gadolinium concentration results for the experimental solutions by the three methods is given in Table D.6. The data in the last column are the recommended values based on the results using the second method. The values include the estimated impurity concentration of gadolinium, 0.002 g/liter. These recommended values are given in Table 3.1 of this report.

**TABLE D.1** Analysis of Gadolinium Concentrations Based on ICP and Loss on Ignition Estimates of Fraction of Gadolinium in Gadolinium Nitrate Powder

Conc. Gd Solution Number	Mass of Materials Used to Prepare Conc. Gd Solution				Amounts Added to Bulk Solution (a)			Bulk Solution Number
	Water (g)	Nitric Acid (g)	Gd Nitrate Powder (g)	Total (g)	Gd <sup>(b)</sup> (g)	Volume (liter)	Gd <sup>(b)</sup> Conc. (g/liter)	
C1	975	46.1	295.29	1316.4	107.69	1.1029	0.501	B-1
C2	781	37.0	298.00	1116.0	108.53	0.9052	1.001	B-2
C3	781	37.0	300.19	1118.2	109.32	0.9041	1.501	B-3
C4	781	37.0	302.41	1120.4	110.13	0.9032	2.000	B-4
C5	781	37.0	124.48	942.5	45.31	0.8422	2.200	B-5

(a) Given amounts are those added to the bulk solution after subtracting quantities in the 7ml analytical samples.

(b) Calculated values.

**TABLE D.2** Analytical Data for Concentrated Gadolinium Solutions

<u>Conc. Gd. Solution Number</u>	<u>Gd Conc. (g/liter)</u>	<u>Density (g/cm<sup>3</sup>)</u>
C1	95.91	1.1860
C2	116.14	1.2234
C3	118.52	1.2273
C4	121.37	1.2310
C5	53.30	1.1099

**TABLE D.3** Analysis of Gadolinium Concentrations Based on Analytical Results of Concentrated Solutions

<u>Bulk Solution Number</u>	<u>Bulk Solution Gd Conc. (g/liter)</u>
B1	0.492
B2	0.976
B3	1.466
B4	1.964
B5	2.161

**TABLE D.4** Recovery Data for ICP Analysis

<u>Sample Number</u>	<u>Gd Added (<math>\mu</math>g)</u>	<u>Gd Recovered (<math>\mu</math>g)</u>	<u>Fraction Recovered</u>
1235	0	1.8	-
1235	500	414.5	0.829
1235	1000	785	0.785
1235	1500	1330	0.887
1235	2000	1670	0.835

**TABLE D.5** Analysis Results of Gadolinium Concentrations Using ICP Method

<u>Bulk Solution Number</u>	<u>Sample Number</u>	<u>ICP Gd Conc. (g/liter)</u>	<u>Actual Gd Conc. (g/liter)</u>
B1	1236	0.421	0.509
B2	1237	0.870	1.035
B3	1238	1.297	1.535
B4	1239	1.685	1.989
B5	1248	1.865	2.200

**TABLE D.6** Summary of Gadolinium Concentration Evaluations

<u>Bulk Solution Number</u>	<u>Sample Number</u>	<u>Method 1<sup>(a)</sup> (g/liter)</u>	<u>Method 2<sup>(a)</sup> (g/liter)</u>	<u>Method 3 (g/liter)</u>	<u>Recommended Gd Conc. (g/liter)</u>
B1	1236	0.501	0.492	0.509	0.49
B2	1237	1.001	0.976	1.035	0.98
B3	1238	1.501	1.466	1.535	1.47
B4	1239	2.000	1.964	1.989	1.97
B5	1248	2.200	2.161	2.200	2.16

(a) Methods 1 and 2 are based on gadolinium additions and do not include the residual 0.002 g/liter Gd. The recommended values include the residual gadolinium.

APPENDIX E

CHEMICAL ANALYSIS DATA OF THE IMPURITIES IN PLUTONIUM-URANIUM NITRATE SOLUTIONS

## APPENDIX E

### CHEMICAL ANALYSIS DATA OF THE IMPURITIES IN PLUTONIUM-URANIUM NITRATE SOLUTIONS

The concentrations of impurity elements in the plutonium-uranium nitrate solutions were determined by analysis with the Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) method. The uncertainty in the ICP results is approximately  $\pm 25\%$ . The ICP analysis results for sample 1215 which cover experiment 107 are given in Table E.1. The ICP results for sample 1215 can be used to estimate the impurity concentrations in the other experiments (i.e., 106, 106R, 109, 110, 110R and 117) by multiplying the results by the ratio of plutonium concentrations.

The ICP analysis results for samples 1235 A and B cover experiments 111 - 116.

**TABLE E.1 Inductively Coupled Plasma  
Spectroscopy Analyses (mg/liter)**

<u>Element</u>	<u>Sample No. 1215</u>	<u>Sample No. 1235A</u>	<u>Sample No. 1235B</u>
Al	68.7	118	126
B	3.0	7.95	10.5
Ba	2.0	3.55	4.30
Ca	41.0	65.4	72.9
Cd	4.3	6.20	5.65
Ce	4.2	6.50	8.00
Cr	57.1	90.5	90.5
Cu	12.0	15.2	18.4
Dy	0.8	0.8	0.8
Fe	246.7	383	379
Gd	<1	(a)	(a)
K	5.0	15	10
La	0.7	(.6)	(.3)
Li	0.2	(a)	(.5)
Mg	8.6	19.0	19.9
Mn	11.4	17.4	17.6
Mo	1.2	(a)	(a)
Na	20.6	40	46
Nd	1.3	(2.1)	(1.4)
Ni	41.4	64	64
Rh	14.0	(a)	(a)
Ru	4.2	(a)	(a)
Si	16.5	30.9	52.0
Sr	0.2	(.35)	(.35)
Te	(a)	(a)	(a)
Ti	17.4	26.7	27.3
Zn	3.6	8.05	8.40
Zr	3.9	6.20	6.10

(a) Element not detected.

Values given in parentheses are approaching detection limit.

APPENDIX F

CHEMICAL ANALYSIS DATA OF THE REFLECTOR WATER SAMPLES

## APPENDIX F

### CHEMICAL ANALYSIS DATA OF THE REFLECTOR WATER SAMPLES

The sample for experiment 106 also covers experiment 106R. The sample for experiment 110 also covers experiment 110R. Reflector water analysis for experiment 117 was not performed; the data would be similar to other experiments.



TABLE F.1 Water Sample Analyses of the Reflector Water



HANFORD ENVIRONMENTAL  
HEALTH FOUNDATION

CO 12325

February 11, 1988

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

Attn: R. C. Lloyd

WATER SAMPLES ANALYSES

The results of the analyses of the seven water samples received January 6, 1988, are attached. Analyses were done in accordance with Standard Methods for the Analysis of Water and Wastewater, 16th ed.

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

*P. A. Thurman*

P. A. Thurman  
Environmental Health Sciences

jt

Attach.

P. O. BOX 100. RICHLAND, WASHINGTON 99352

TABLE F.1 (Contd)

<u>Parameter</u>	<u>Exp. 106, 107, 108</u>	<u>Exp. 109, 110</u>	<u>Exp. 111</u>	<u>Exp. 112, 113</u>	<u>Exp. 114</u>	<u>Exp. 115</u>	<u>Exp. 116</u>
pH	7.0	7.0	7.5	7.2	7.3	7.1	7.4
Total alkalinity (mg/L)	55	55	56	56	54	57	56
HCO <sub>3</sub> alkalinity (as CaCO <sub>3</sub> ) (mg/L)	50	50	56	52	50	52	51
CO <sub>3</sub> alkalinity (as CaCO <sub>3</sub> ) (mg/L)	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
Total dissolved solids (mg/L)	86	85	89	79	76	101	105
Fluoride (mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Chloride (mg/L)	0.91	0.91	0.92	0.97	0.98	0.95	0.94
Nitrate (as N) (mg/L)	<0.05	<0.05	.088	<0.05	<0.05	<0.05	<0.05
Sulfate (mg/L)	10.6	10.7	10.7	10.7	11.7	11.5	10.5
Cadmium (mg/L)	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005	<0.0005
Copper (mg/L)	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Chromium (mg/L)	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Iron (mg/L)	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Lead (mg/L)	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Manganese (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zinc (mg/L)	0.08	0.06	0.08	0.07	0.42	0.38	0.08

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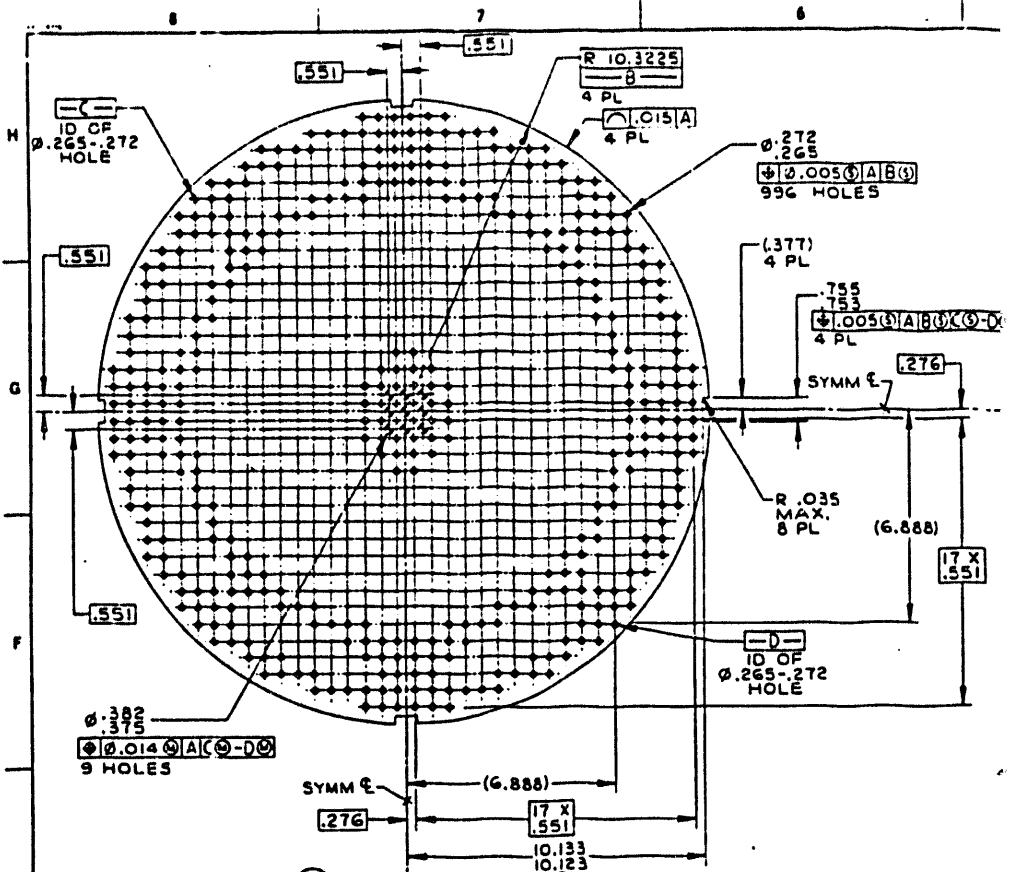
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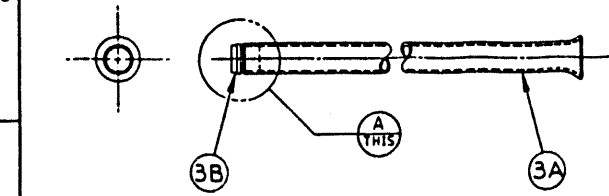
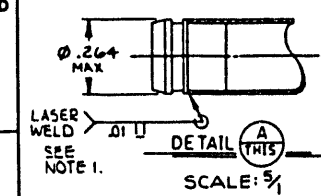




① UPPER BAFFLE  
 MATL: SST 304L  
 SEE NOTE 1  
 SCALE: 1/2

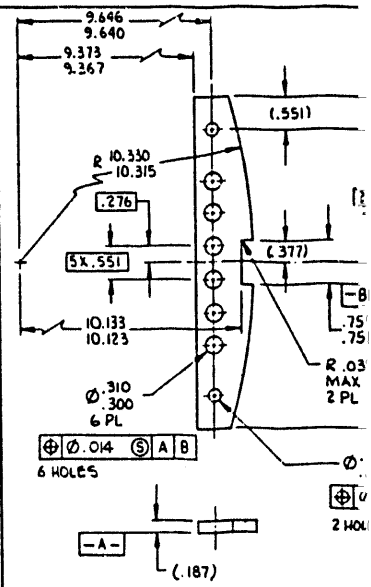
NOTES:  
 1. MATERIAL SHALL CONFORM TO ASTM A240. SEE NOTE 2 ON DWG NO. X3E1279.

ITEM NO.	QUANTITY	DESCRIPTION	UNIT	ITEM NO.	QUANTITY	DESCRIPTION	UNIT
3	1	TUBE WELDMENT		3A	6	TUBE	
3B	1	PLUG		3B	5	PLUG	



③ TUBE WELDMENT  
 SCALE: 5/8

NOTES:  
 1. INSPECT AND ACCEPT ACCORDING TO SPEC # SP-BT-008-FI-MMA SECTION 05520 3.02-B.



⑤ SPACER HOLDER  
 MATL: SST 304L  
 187 THK CR BAR  
 SCALE: FULL

NOTES:  
 1. MATERIAL SHALL CONFORM TO ASTM A276. SEE NOTE 2 ON DRAWING NO. X3E1279-0000

ITEM & SECTION KEY

1	SECTION	DISAG
2	SECTION	DISAG
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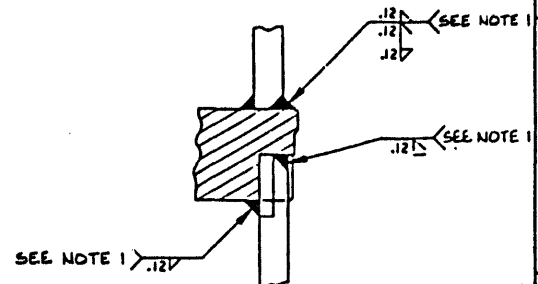
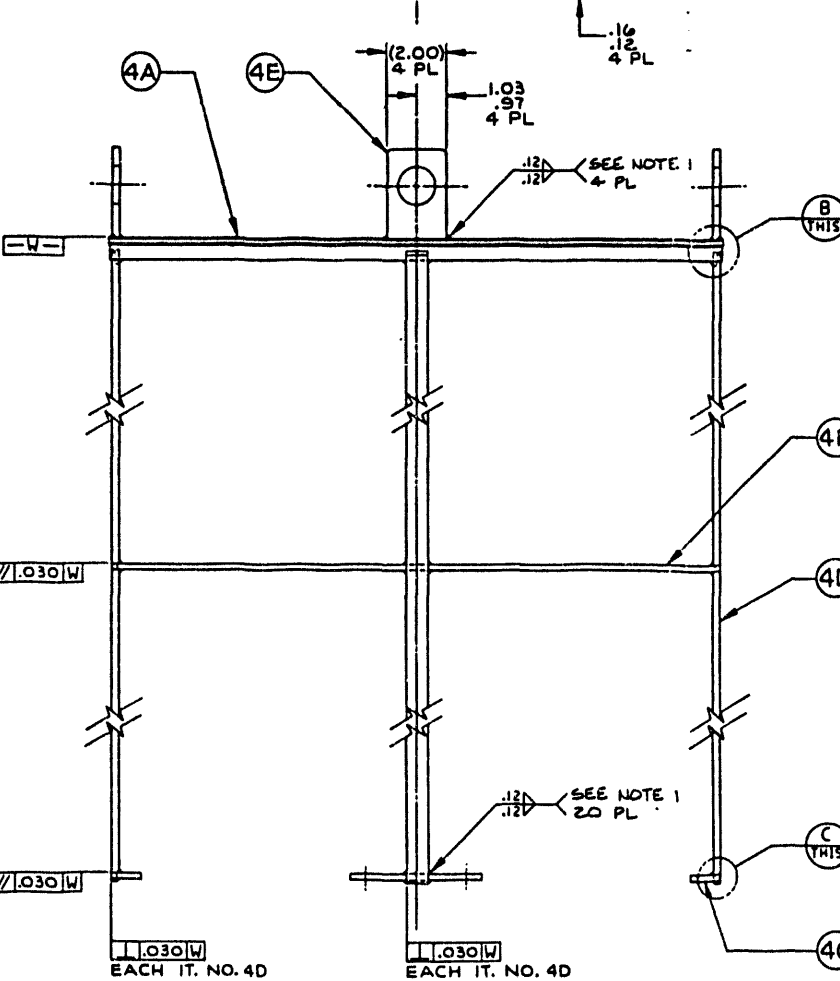
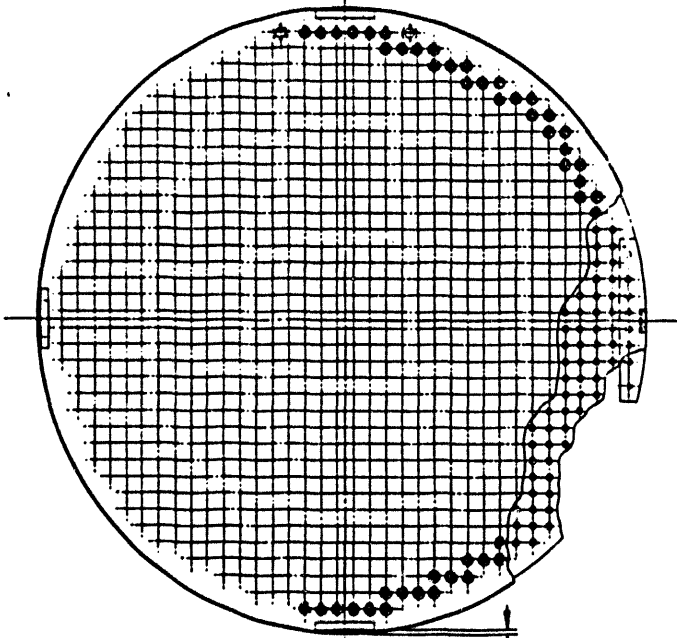
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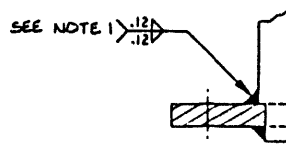
ITEM NO.	DESCRIPTION	QTY	UNIT	REVISION	DATE
4	TOP / BAFFLE PL WELDMENT	1			
4A	TOP PLATE	1			
4B	UPPER BAFFLE	1			
4C	SPACER HOLDER	5			
4D	SIDE BAR	4			
4E	LIFTING LUG	2			

**NOTES:**

1. VISUALLY AND LIQUID PENETRANT INSPECT ACCORDING TO SPEC. #SP-B7-008-F1-MMA SECTION 05520 PART 3.02-A.



**DETAIL B**  
SCALE: 2/1  
TYP 4 PLACES



**DETAIL C**  
SCALE: 2/1  
TYP 4 PLACES EACH ON ITEM NO. 4B & 4C

**4** TOP / BAFFLE PL WELDMENT  
SCALE: 1/2

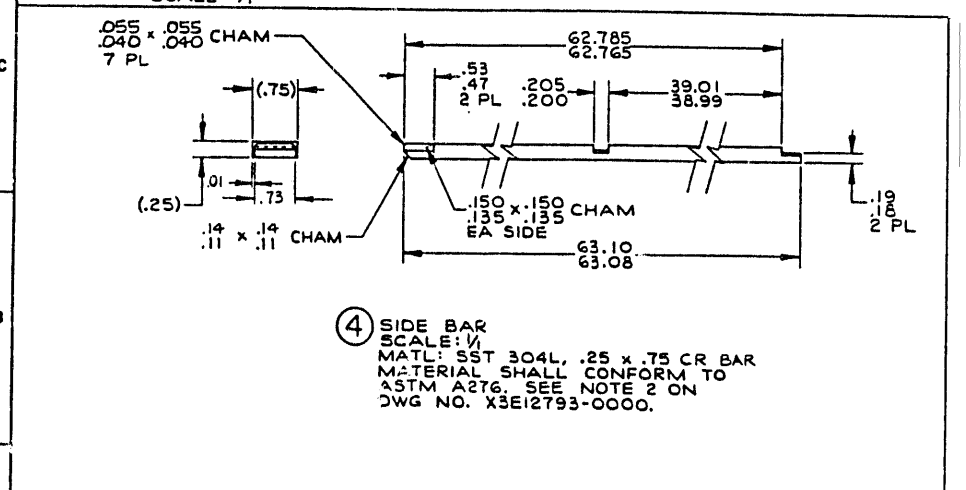
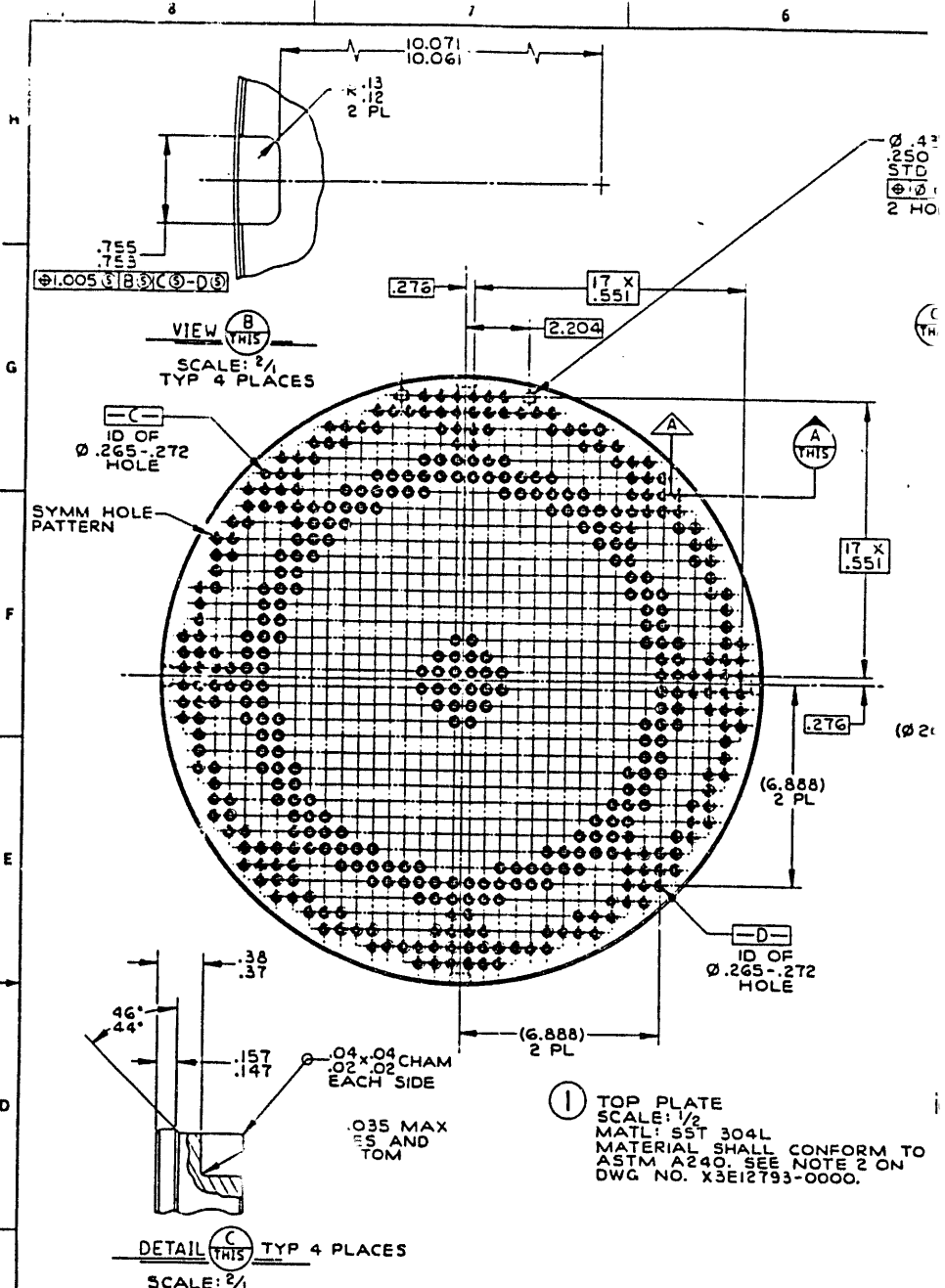
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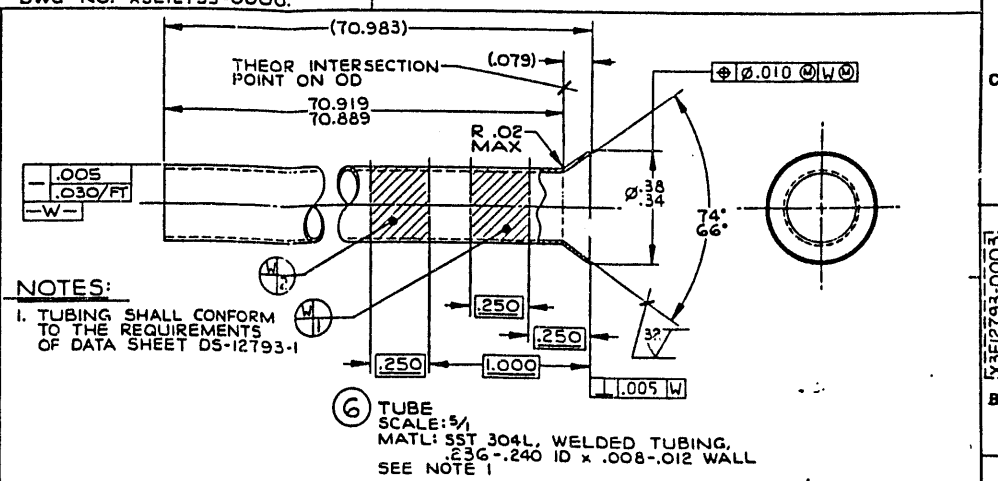
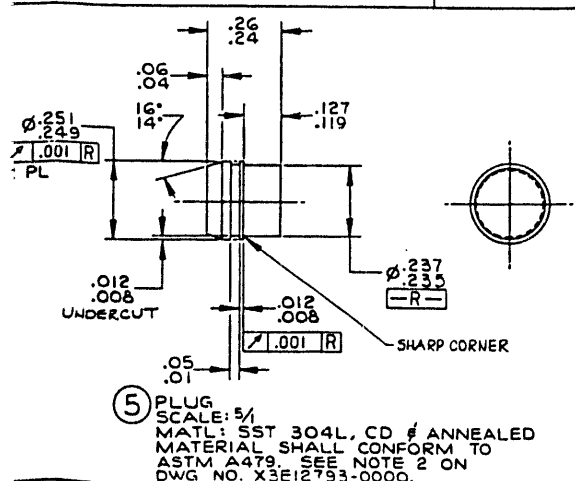
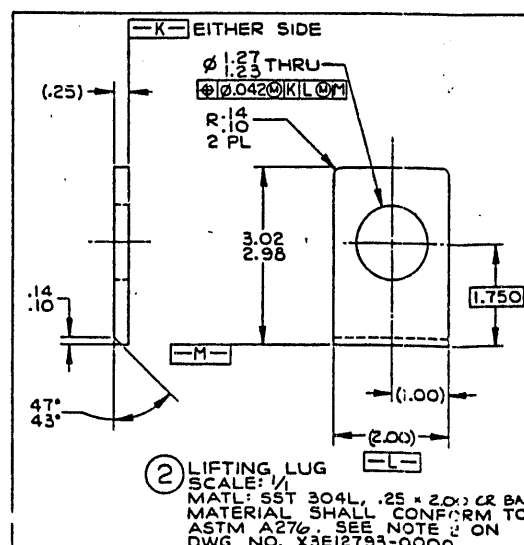
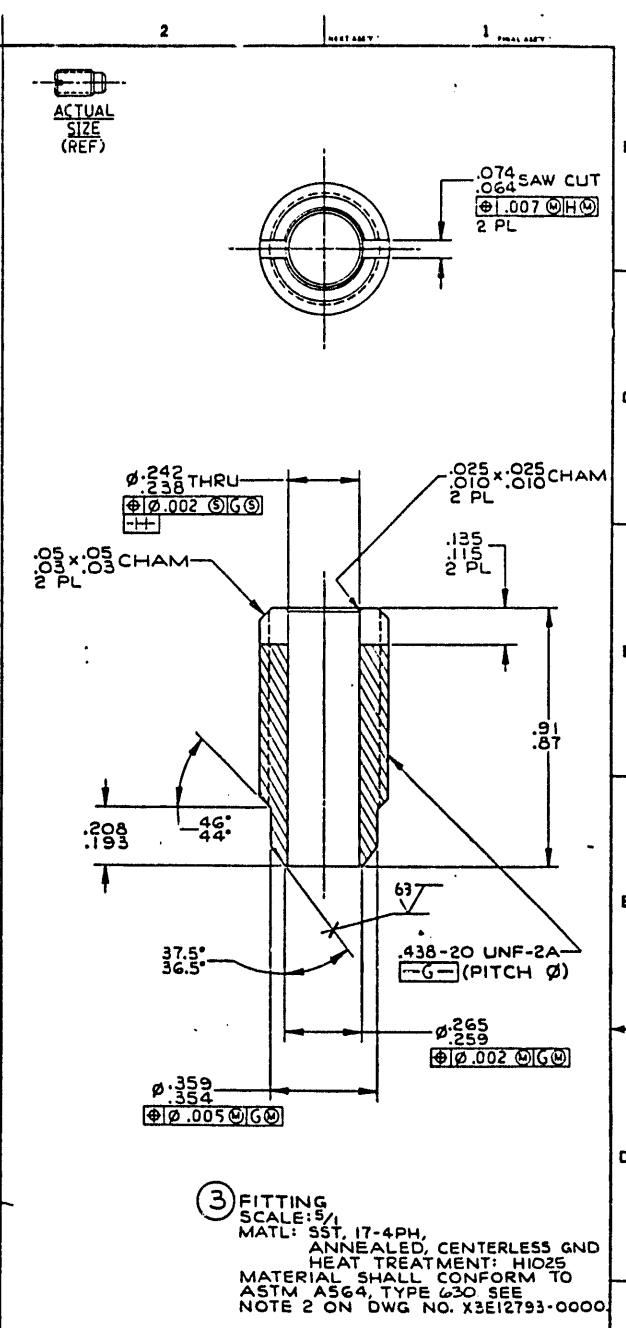
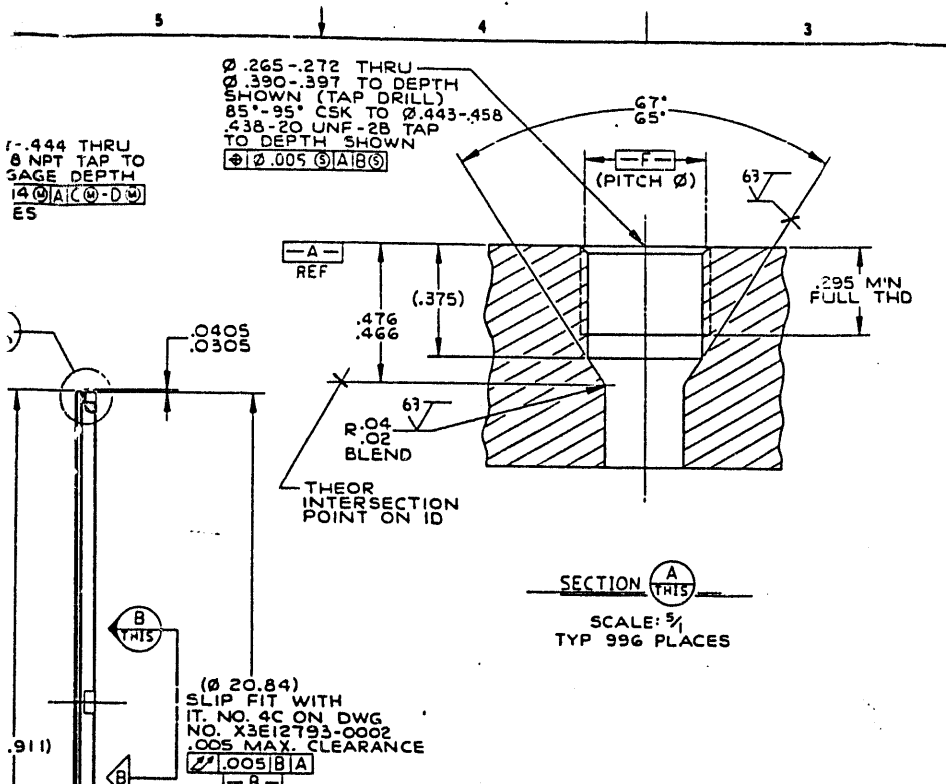


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3	SECTION 0130	SECTION 0130
4	SECTION 0140	SECTION 0140
5	SECTION 0150	SECTION 0150
6	SECTION 0160	SECTION 0160
7	SECTION 0170	SECTION 0170
8	SECTION 0180	SECTION 0180
9	SECTION 0190	SECTION 0190
10	SECTION 0200	SECTION 0200

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