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APPLIED TECHNOLOGY

Criticality Experiments with Mixed Plutonium and Uranium Nitrate Solution at a Plutonium Fraction of 0.5 in Annular Cylindrical Geometry

R. C. Lloyd

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

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Work performed for the Consolidated
Fuel Reprocessing Program and the
Power Reactor and Nuclear Fuel Development
Corporation of Japan under the Joint
Memorandum of Agreement on Criticality
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 0.5. These experiments were a part of the Criticality Data Development Program between the United States Department of Energy (USDOE), and the Power Reactor and Nuclear Fuel Development Corporation (PNC) of Japan. A complete description of, and data from, the experiments are included in this report. The experiments were performed with mixed plutonium-uranium solutions in annular cylindrical geometry. The measurements were made with a water reflector. The central region included a concrete annular cylinder containing B_4C. Interior to the concrete insert was a stainless steel bottle containing plutonium-uranium solution. The concentration of the solution in the annular region was varied from 116 to 433 g (Pu + U)/liter. The ratio of plutonium to total heavy metal (plutonium plus uranium) was 52% for all experiments.

CONTENTS

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CRITICALITY EXPERIMENTS WITH MIXED PLUTONIUM AND URANIUM NITRATE SOLUTION AT A PLUTONIUM FRACTION OF 0.5 IN ANNULAR 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, 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 fast breeder reactor 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 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, 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, 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.

This report contains a description of, and data from, the criticality experiments conducted with mixed plutonium-uranium solutions at Pu/(Pu + U) ratios of approximately 0.5. The experiments were performed in annular cylindrical geometry. The measurements were made with a water reflector. The central region included a concrete annular cylinder containing B_4C. Interior to the concrete insert was a stainless steel bottle containing plutonium-uranium solution. The concentration of the solution in the annular region was varied from 116 to 433 g (Pu + U)/liter.

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 annular cylinder 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.

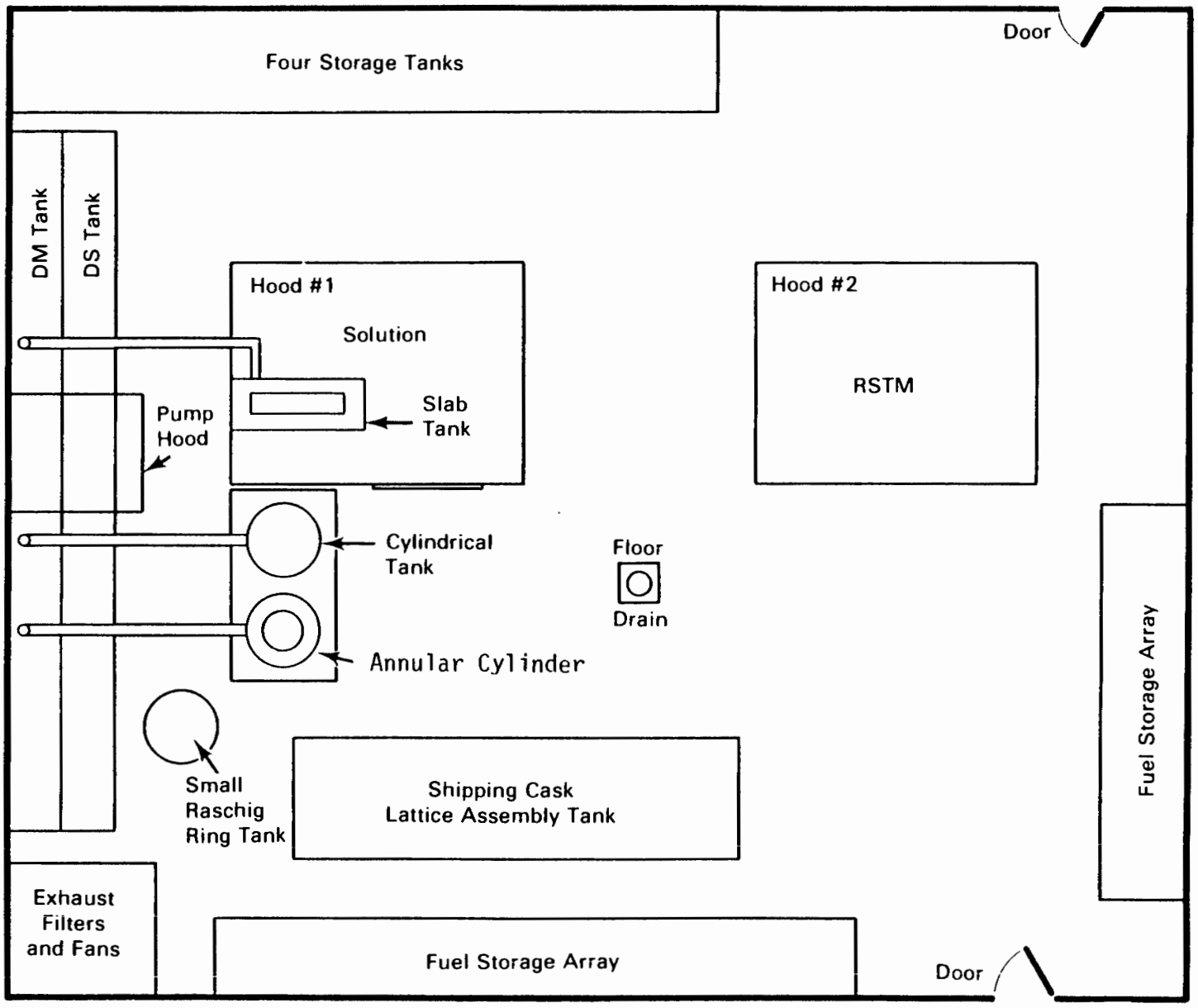


FIGURE 2.1 Floor Plan Schematic of the Critical Assembly Room

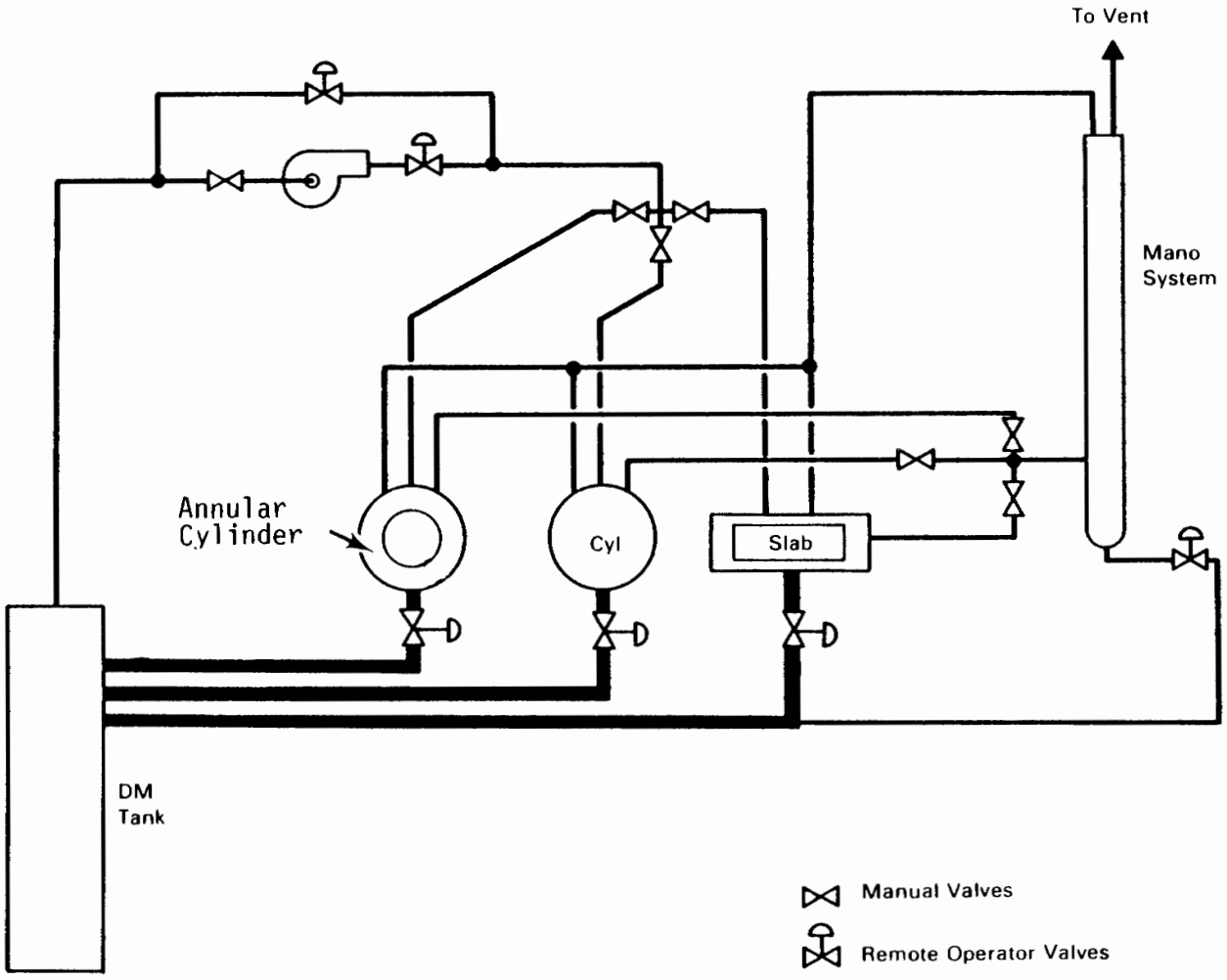


FIGURE 2.2 Piping Schematic for the Three Experimental Vessels

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 in this report was an annular cylinder and it was located on the left side of the system. The control and safety blade mechanisms are 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 tank and for visual inspection. This reflector tank was fabricated of carbon steel (CS). A schematic of the experimental vessel arrangement giving dimensions is shown in Figure 2.4.

The annular cylindrical vessel was fabricated of Type 304L stainless steel. The vessel had an outside diameter of 53.3 cm and an inside diameter of 25.4 cm. The wall thickness was 0.074 cm. The control and safety blades are external to the vessel and are withdrawn during the neutron flux determination during the critical approach measurement. A schematic of the annular cylindrical vessel is shown in Figure 2.5 and a photograph of the top of the system is shown in Figure 2.6. This shows the annular concrete insert and the bottle of solution located in the central region. Figure 2.7 is a photograph showing the annular tank and bottle insert prior to installation for critical experiments. A schematic of the concrete insert is shown in Figure 2.8. The figure includes as-built dimensions for the inside and outside diameters of the insert, as well as other dimensions based on the fabrication specifications.

The experiments with the annular cylinder were conducted with the reflector tank containing water. The reflector tank was filled to the level 24.8 cm below the top of the annular vessel. The distance between the annular cylinder (portion below the annular region) and the bottom of the reflector tank (inside surface) is 40.1 ± 0.1 cm.

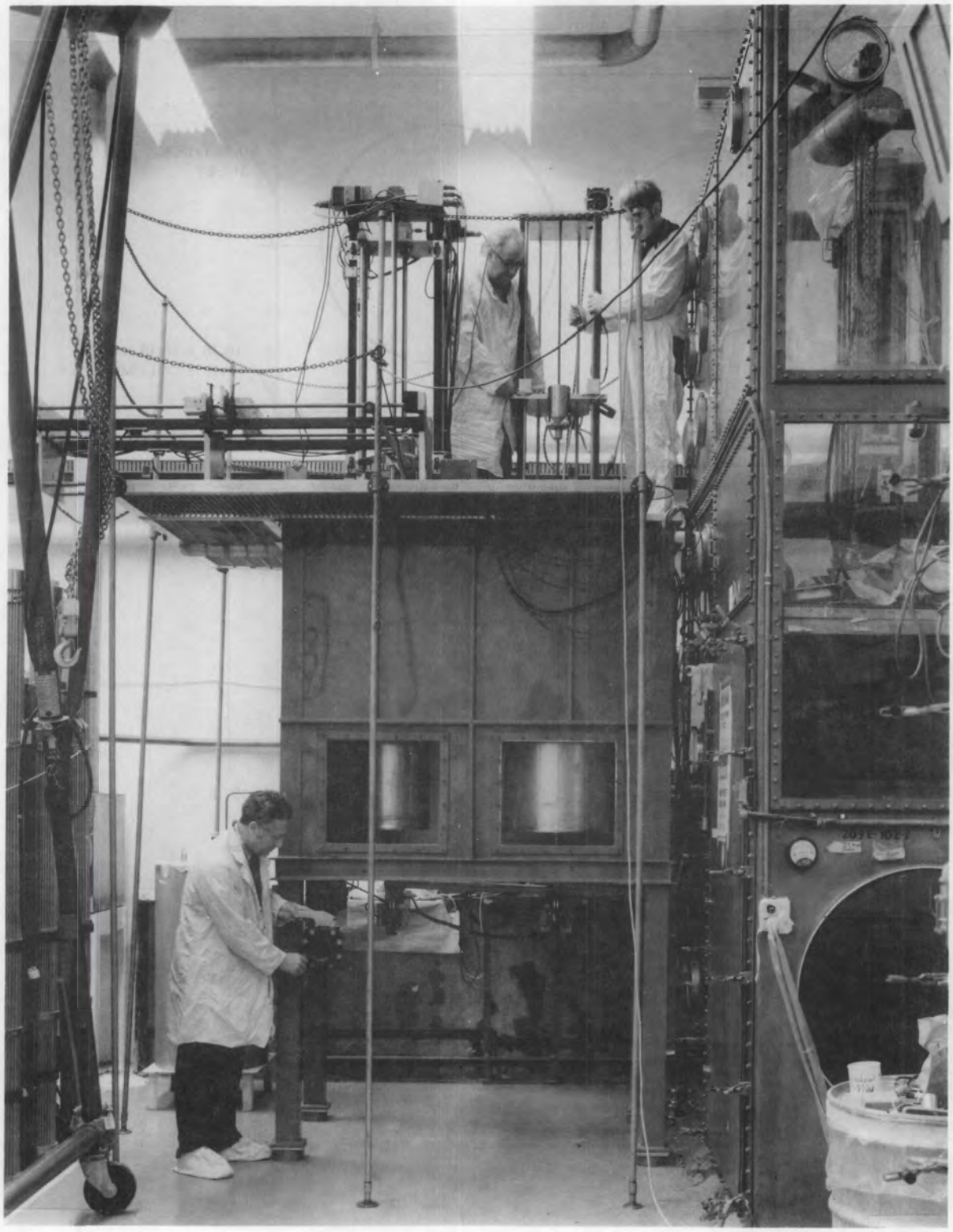


FIGURE 2.3 Photograph of the Cylindrical Vessel System

ORNL–DWG 86–11826A

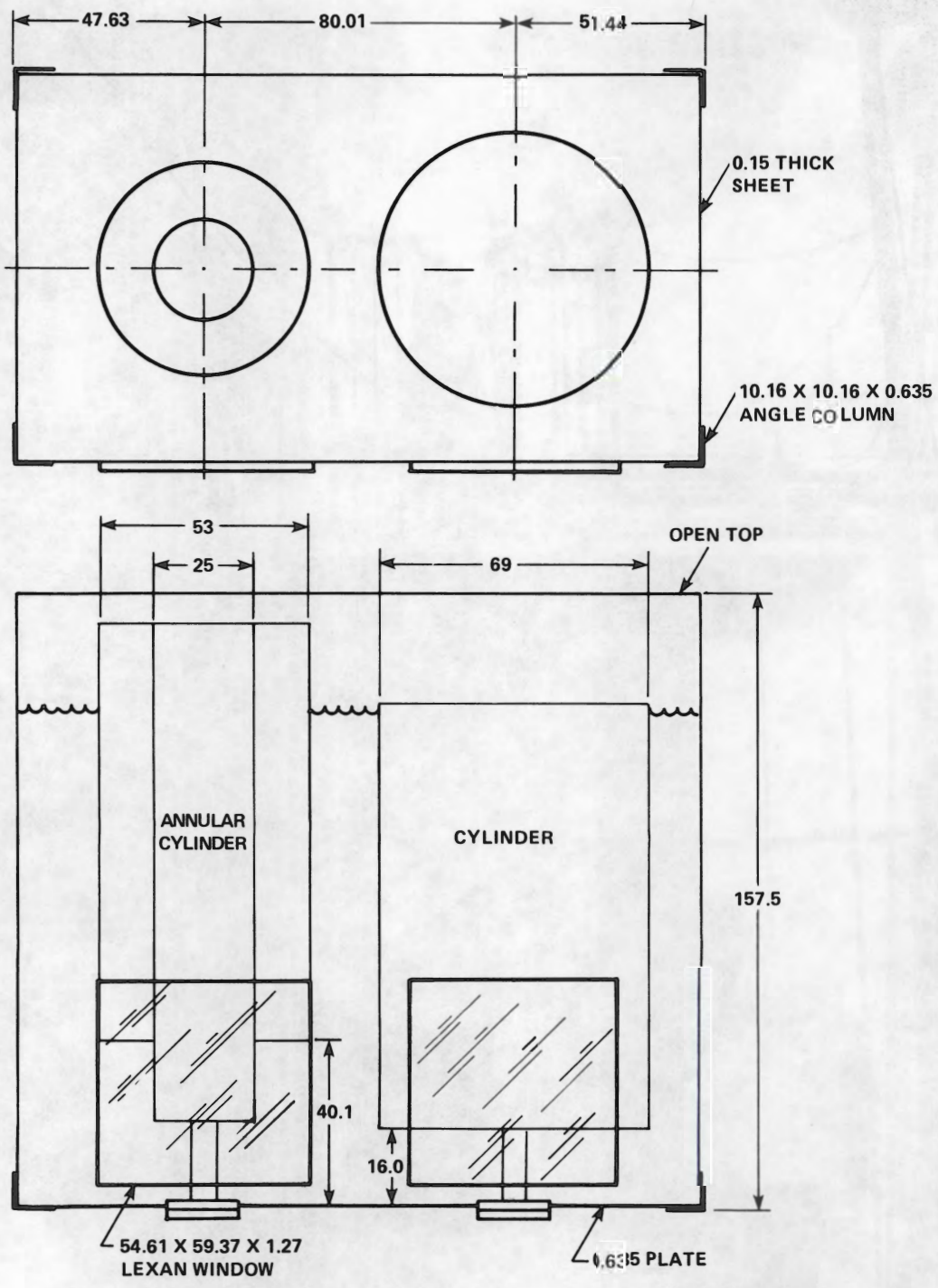


FIGURE 2.4 Schematic of Dual Cylinder Tank with Annular and Large Cylinder Mounted

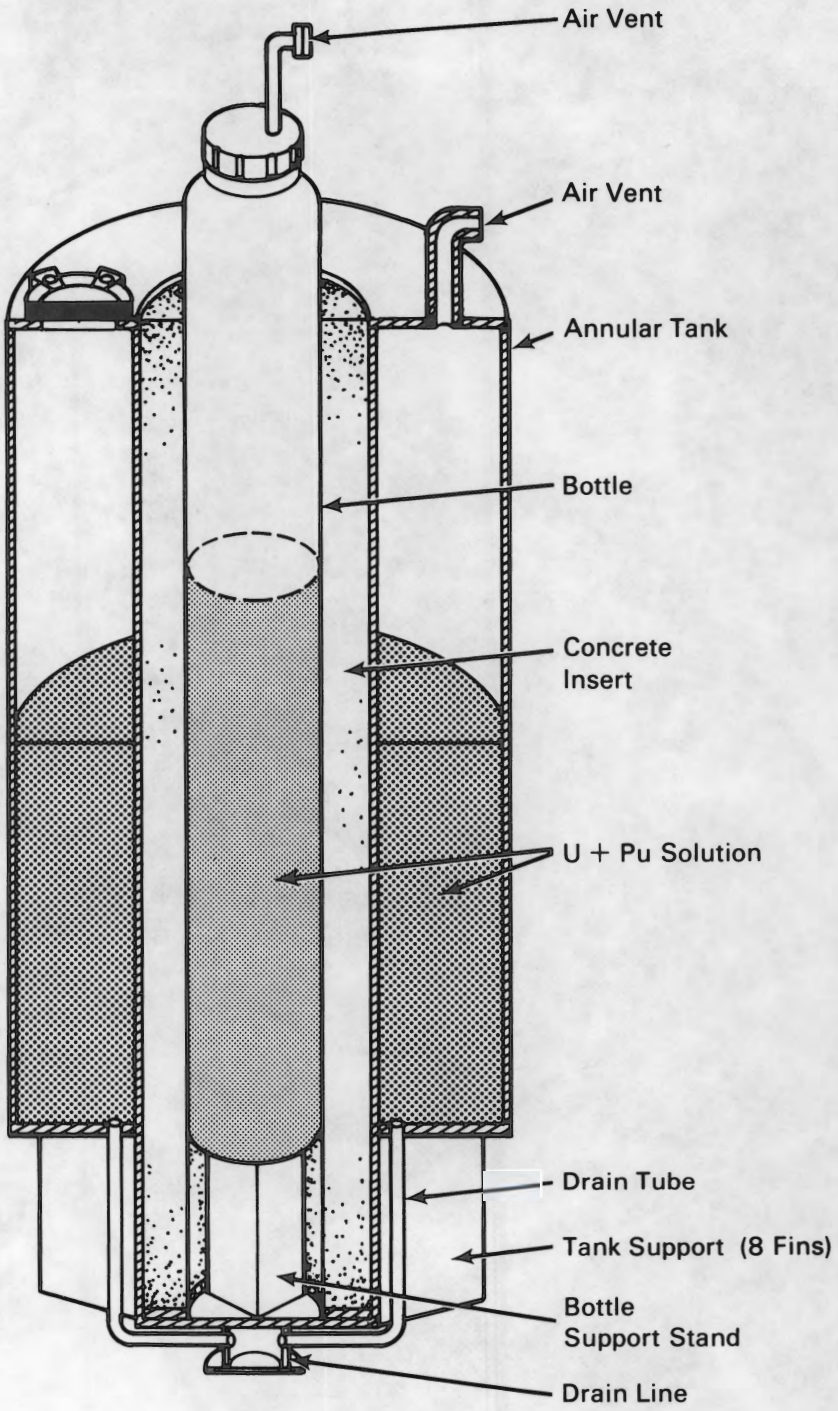


FIGURE 2.5 Schematic of the Annular Cylindrical Vessel with Concrete Insert and Bottle

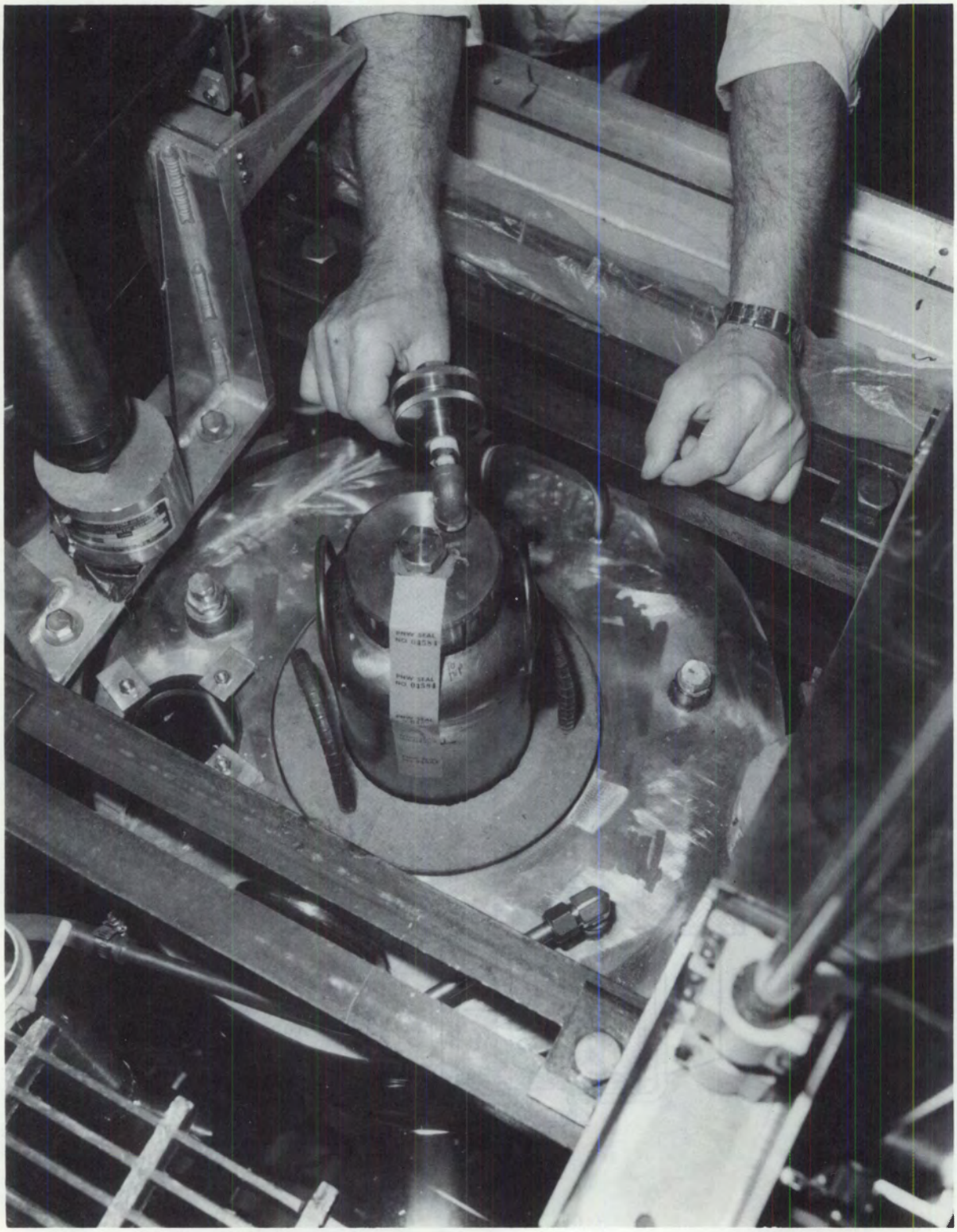


FIGURE 2.6 Photograph of the Annular Assembly

FIGURE 2.7 Photograph Showing Annular Tank and Bottle

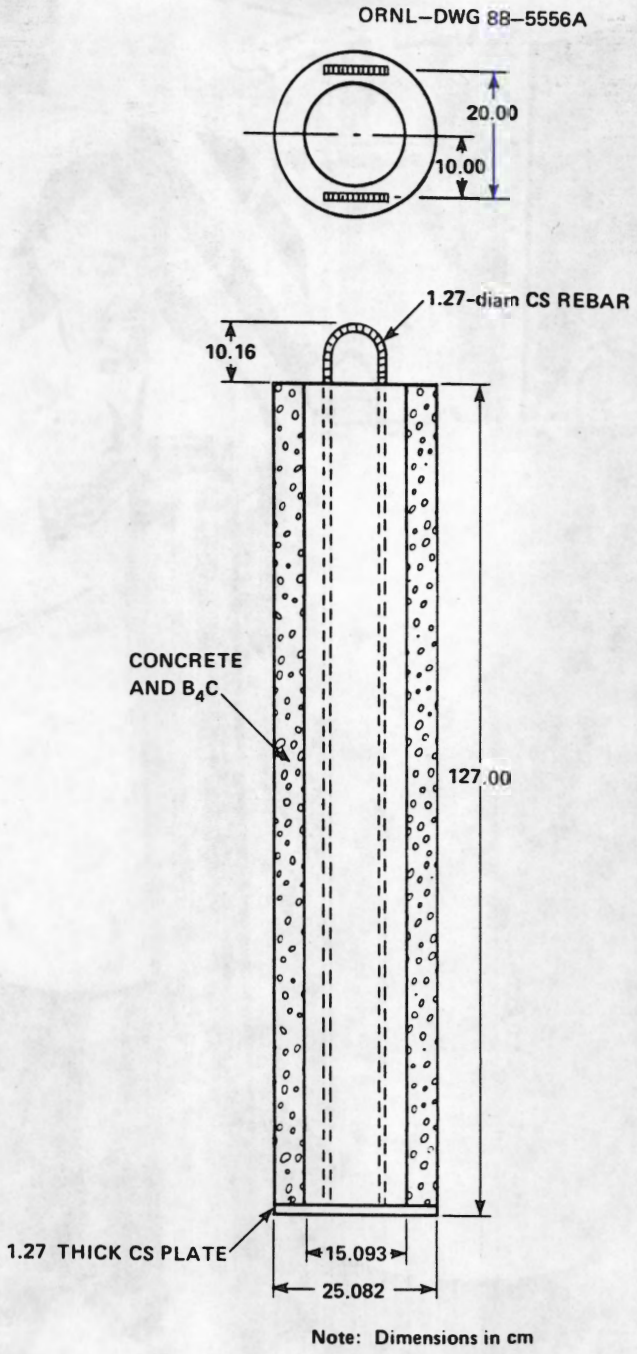


FIGURE 2.8 Schematic of Annular Concrete Insert

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 are provided in Appendix A for the cylindrical vessel system.

Engineering drawings are provided in Appendix B for the annular tank assembly.

3.0 EXPERIMENTAL RESULTS

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

3.1 CRITICALITY MEASUREMENT TECHNIQUES

The critical heights for the experiments reported herein were determined using the critical approach method (Clayton 1985). In this critical approach method, neutron flux measurements are made as the height of solution is incrementally increased. Inverse count rate is plotted versus solution height. 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 height in each experimental assembly, are included in Appendix C.

3.2 CRITICALITY DATA

The criticality data for this report were obtained from August 1985-January 1986, when five experiments were completed using the annular cylindrical vessel. The data are summarized in Table 3.1. The chemical analysis and data for sample #1152 for the plutonium concentration includes Pu^{+6} since this material had been recently concentrated. The value for the free acid was corrected to the date of the experiment (1/29/86). The plutonium concentration data for samples 1110, 1111, 1112, and 1122 were adjusted to remove a positive bias which resulted when the samples were not ion exchanged in the original analyses. Further discussion on the method of adjusting these concentration data is provided in Appendix D. 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

TABLE 3.1 Criticality Measurements with (Pu + U) Nitrate Solution in the Annular Cylinder[a]

Run Date	Case Number	Experiment Number	Reflector	Sample Number	Pu (g/liter)	U (g/liter)	Density[b] (g/cm^3)	Free Acid (M)	Critical Height (cm)
08/09/85	4	052	Water	1111	172.6[c]	156.41	1.5295	1.07	31.32
08/13/85	4	052R	Water	1111	172.6[c]	156.41	1.5295	1.07	31.06
08/19/85	5	053	Water	1112	113.0[c]	102.55	1.3569	0.88	29.24
09/12/85	9	057	Water	1122[d]	60.7[c]	55.62	1.1957	0.53	31.05
01/29/86	12	062	Water	1152	226.22	207.09	1.6752	(1.41)[e]	34.49

(a) Annular cylinder with 2% B_4C concrete insert and bottle; material contained in inner bottle (Sample Number 1110): 6.53 ± 0.02 liters of (Pu + U) nitrate solution, the Pu concentration was 76.2 g/liter, the U concentration was 69.24 g/liter, the density was 1.2483 g/cm^3 and the H^+ concentration was 0.81 M. The bottle was filled to 39.3 ± 0.2 cm.

(b) Density measured at 23°C

(c) Appendix D provides discussion regarding how these data were obtained.

(d) Samples 1122 and 1121 were of the same solution mixture. Sample 1122 data reported here since it was taken closer in the time to the experiment.

(e) Sample value for 1152 corrected to 01/29/86 (H^+ = 1.41 M, Pu^{+6} = 52.3 g/liter).

Company, Hanford Engineering Development Laboratory (HEDL) from samples of solution supplied to them. The sample analyses methods and descriptive titles are given in Table 3.2. The critical heights were calculated by a least squares fit to the inverse neutron multiplication data from three neutron detectors. (Computer printout provided in Appendix C). The ^{241}Am content for each sample analyzed, the analysis date and the experiments covered by that sample are given in Table 3.3. The isotopic analyses values for the plutonium and uranium of 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 E 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 F.

The composition of the concrete insert used in these experiments is given in Appendix G. Also provided are sample analyses for the boron and certificate of analysis provided by the vendor for the B_4C. Weight changes of the sample of concrete are also given in Appendix G. The weights of the ingredients used to make up the concrete insert are given.

3.3 SOURCES OF ERROR

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

TABLE 3.2 Chemical Analyses Methods

Measurement	Method Title		Date of Approval
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.

TABLE 3.3 Chemical Analysis Values for Americium-241

Sample Number	^{241}Am (ug/ml)	Analysis Date
1110	394	09/16/85
1111	902	10/21/85
1112	577	09/16/85
1121	302	09/23/85
1152	1193	05/16/86

Sample 1110 is the analysis for solution in the bottle

Sample 1111 covers experiments 052 and 052R

Sample 1112 covers experiment 053

Sample 1121 covers experiment 057

Sample 1152 covers experiment 062

TABLE 3.4 Isotopic Analyses Values of Pu and U[a]

	Sample 1110[b]	Sample 1111[c]	Sample 1112[d]	Sample 1121[e]	Sample 1152[f]
Pu[g]	09/10/85	10/24/85	09/10/85	09/25/85	05/14/86
238	0.029 ± 0.001	0.024 ± 0.002	0.029 ± 0.001	0.027 ± 0.001	0.028 ± 0.001
239	91.03 ± 0.04	91.12 ± 0.04	91.09 ± 0.04	91.10 ± 0.04	91.10 ± 0.04
240	8.38 ± 0.04	8.30 ± 0.04	8.33 ± 0.04	8.31 ± 0.04	8.33 ± 0.04
241	0.473 ± 0.003	0.463 ± 0.003	0.469 ± 0.003	0.467 ± 0.002	0.449 ± 0.004
242	0.098 ± 0.004	0.092 ± 0.002	0.093 ± 0.004	0.092 ± 0.002	0.090 ± 0.002
U[h]	09/17/85	10/25/85	09/17/85	09/24/85	05/13/86
238	99.264 ± 0.006	99.263 ± 0.005	99.264 ± 0.006	99.266 ± 0.006	99.257 ± 0.005
236	0.023 ± 0.001	0.024 ± 0.002	0.022 ± 0.001	0.022 ± 0.001	0.027 ± 0.002
235	0.705 ± 0.005	0.705 ± 0.004	0.706 ± 0.005	0.702 ± 0.005	0.707 ± 0.005
234	0.009 ± 0.002	0.009 ± 0.002	0.008 ± 0.002	0.009 ± 0.003	0.010 ± 0.002

(a) All values given in wt%

(b) Sample 1110 is for the solution in the bottle

(c) Sample 1111 is for Experiments 052 and 052R

(d) Sample 1112 is for Experiment 053

(e) Sample 1121 is for Experiment 057

(f) Sample 1152 is for Experiment 062

(g) Date of Pu analysis

(h) Date of U analysis

TABLE 3.5 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)
	CAR	DM Tank	Reflector		
Ø52	2Ø.9	22.Ø	19.9	24.8	22.2
Ø52R	2Ø.3	23.3	19.1	24.8	22.2
Ø53	29.1	25.1	25.6	24.8	22.2
Ø57	19.9	23.9	19.9	24.8	22.2
Ø62	22.4	19.3	18.4	24.8	22.2

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 3.6 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

The uncertainty values for the chemical analyses was provided by M. C. Burt of the Chemical and Analysis Section of the Westinghouse Hanford Company, Hanford Engineering Development Laboratory. The critical height uncertainty is given as 1.6 mm though the least square fitting of approach data for three counting systems would indicate a smaller value as reasonable. The 1.6 mm is the smallest unit on the sight tube.

4.0 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

Clayton, E. D. (1985). Neutron Source Multiplication Method, PNL-SA-13357, Pacific Northwest Laboratory, Richland, Washington.

Lloyd, R. C. (1986). Critical Experiments with Mixed Plutonium and Uranium Nitrate Solution at a Plutonium Fraction of 0.5 in Slab and Cylindrical Geometry. PNL-5768, Pacific Northwest Laboratory, Richland, Washington.

Primm, R. T. III, et. al., (1986). Critical Experiments with Mixed Plutonium-Uranium Nitrate Solutions having Pu/(Pu + U) Ratios Greater than 0.5. ORNL-6161, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Ryan, J. L., et. al., (1985). "Preparation of Acid Standards for and Determination of Free Acid in Concentrated Plutonium-Uranium Solutions." Analytical Chemistry. 57:1423.

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_2O Tank and Cover H-2-33856, Sheet 2 of 5

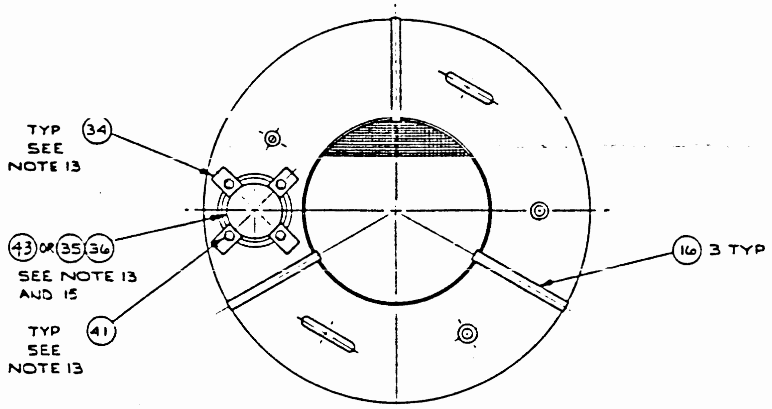
CFRP Process Tanks H-2-33856, Sheet 3 of 5

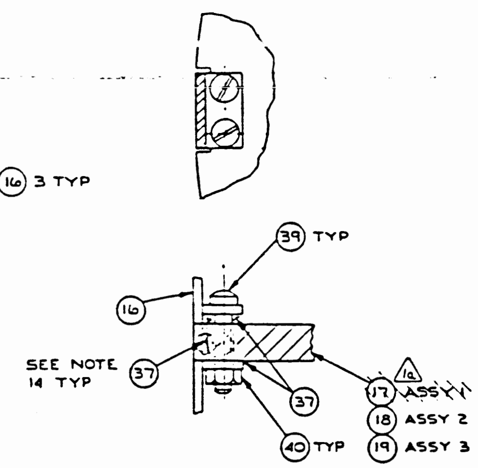
CFRP Tank Covers and Shield H-2-33856, Sheet 4 of 5

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

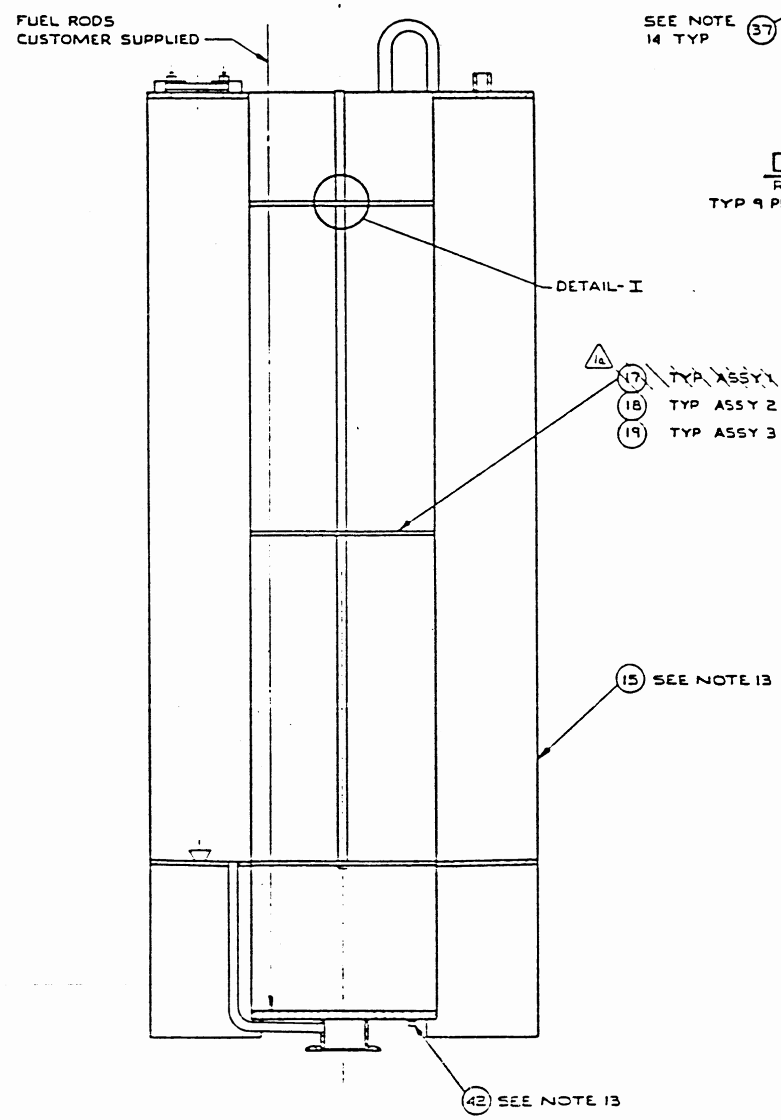
APPENDIX B

ENGINEERING DRAWINGS OF THE ANNULAR TANK ASSEMBLY





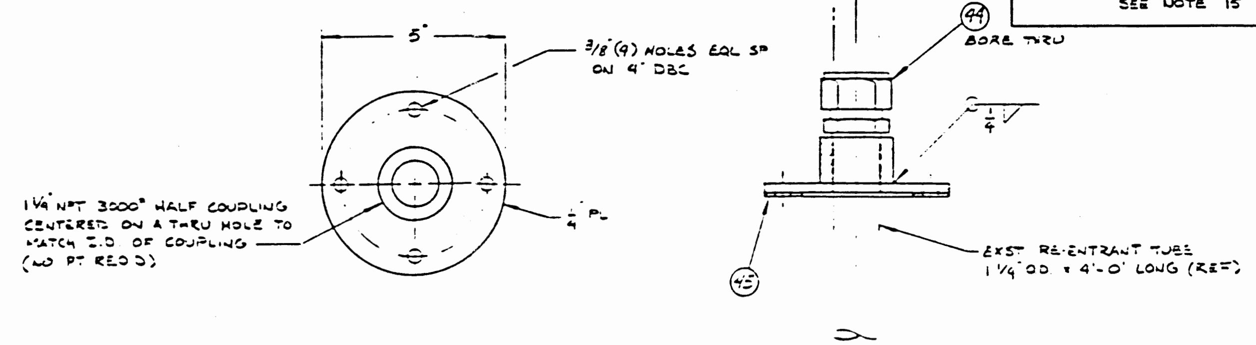
DETAIL - I
ROTATED 90° CCW
TYP 9 PLACES EA IN ASSYS 1,2 AND 3



(1) ASSEMBLY SHOWN

(2) ASSEMBLY AS NOTED

(3) ASSEMBLY SHOWN



(43) RE-ENTRANT TUBE HOLDER
SCALE: NONE
MAT'L: 304 OR 316 STAINLESS STEEL

GENERAL NOTES

(UNLESS OTHERWISE NOTED)

1. ALL DIMENSIONS ARE IN INCHES.
2. DIMENSIONING AND TOLERANCING ARE IN ACCORDANCE WITH ANSI Y14.5 - 1973.
3. REMOVE ALL BURRS AND BREAK ALL SHARP EDGES.
4. ALL MATERIALS SHALL BE AS SPECIFIED OR AN APPROVED EQUAL.
5. [illegible] STEEL WITH APPROVAL OF RESPONSIBLE PNL ENGINEER. STAINLESS STEEL SHALL BE IN ACCORDANCE WITH THE FOLLOWING ASTM SPECIFICATIONS:

PLATES AND SHEETS	ASTM A240
TUBES	ASTM A269
PIPE	ASTM A312

6. TOLERANCES: FRACTIONAL ± 1/16
   .XX ± .01
   .XXX ± .005
   ANGLES ± 3°
7. PN 15 AND 30 SHALL BE HELIUM BUBBLED TESTED @ 4 PSI. REPAIR ALL LEAKS.
8. CERTIFICATION REQUIREMENTS:
   CERTIFIED MATERIAL TEST REPORTS REQUIRED ON SST-BARS, SHEETS AND SHAPES.
   LETTER OF COMPLIANCE OR QC RECEIVING INSPECTION REPORT MAY BE SUBSTITUTED AS LETTER OF COMPLIANCE ON ALL PLASTIC MATERIAL, ENGINEERED PRODUCTS AND CADMIUM SHEET. FURNISH SAMPLES OF MATERIALS PER PNL INSTRUCTIONS.
9. ALL WELDING OF SHEET METAL PER AWS D9.1 - 1980.
10. LIQUID PENETRANT (PT) INSPECTION AND ACCEPTANCE CRITERIA SHALL BE IN ACCORDANCE WITH ANSI B31.1, PARAGRAPH 136.4.4.
11. VESSEL DESIGN PRESSURE = 2.6 PSI AT AMBIENT TEMPERATURE.
12. CONTRACTOR SHALL DOCUMENT AND FURNISH TO CUSTOMER ACTUAL CONCRETE INGREDIENT WEIGHTS AND MIXING PROCEDURES. CUSTOMER WILL FURNISH TO CONTRACTOR CONCRETE FORMULA. CONCRETE SHALL BE VIBRATED TO REDUCE OPEN POCKETS.
13. ONLY ONE OF THE FOLLOWING PART NUMBERS WILL BE NEEDED, AND REUSED IN ASSEMBLIES 1 THRU 14 - PART NUMBERS 15, 35, 36 AND 42. ONLY FOUR OF THE FOLLOWING PART NUMBERS WILL BE NEEDED, AND REUSED IN ASSEMBLIES 1 THRU 14 - PART NUMBERS 34 AND 41. ONLY THE FOLLOWING PART NUMBER WILL BE NEEDED AND REUSED IN ASSEMBLIES 8 THRU 11, 13 AND 14 - PART NUMBER 30. TWO OF THE FOLLOWING PART NUMBERS WILL BE NEEDED AND REUSED IN ASSEMBLIES 8,9,10,11,13 & 14 - PART NUMBERS 30,31 & 38.
14. SHIM AS REQUIRED FOR A BIND FREE INSERTION AND REMOVAL OF FUEL RODS.
15. PARTS 43,44 & 45 REPLACE PARTS 34,35 & 36 WHEN ASSEMBLY IS USED WITH RE-ENTRANT TUBE.

CSA — THIS FLAG IDENTIFIES A CRITICALITY SAFETY DIMENSION WHICH SHALL NOT BE CHANGED WITHOUT APPROVAL OF THE TECHNICAL LEADER, NUCLEAR SAFETY.

QTY														PN	DESCRIPTION	MATL / REF
													X	1	ASSEMBLY	
												X		2	ASSEMBLY	
											X			3	ASSEMBLY	
										X				4	ASSEMBLY	SH 2
									X					5	ASSEMBLY	
								X						6	ASSEMBLY	
							X							7	ASSEMBLY	
						X								8	ASSEMBLY	
					X									9	ASSEMBLY	
				X										10	ASSEMBLY	
			X											11	ASSEMBLY	
		X												12	ASSEMBLY	
	X													13	ASSEMBLY	
X														14	ASSEMBLY	SH 2
SEE NOTE 13														15	TANK	SH 3
-	-	-	-	-	-	-	-	-	-	-	3	3	3	16	LATTICE SUPPORT	SH 3
-	-	-	-	-	-	-	-	-	-	-	-	-	3	17	LATTICE PLATE	SH 4
-	-	-	-	-	-	-	-	-	-	-	-	3	-	18	LATTICE PLATE	
-	-	-	-	-	-	-	-	-	-	-	3	-	-	19	LATTICE PLATE	SH 4
-	-	-	-	-	-	-	-	-	-	1	-	-	-	20	SOLID CONCRETE INSERT W/0% B_4C	SH 5
-	-	-	-	-	-	-	-	-	1	-	-	-	-	21	SOLID CONCRETE INSERT W/1% B_4C	
-	-	-	-	-	-	-	-	1	-	-	-	-	-	22	SOLID CONCRETE INSERT W/3% B_4C	
-	-	-	-	-	-	-	1	-	-	-	-	-	-	23	SOLID CONCRETE INSERT W/10% B_4C	
-	-	-	-	-	-	1	-	-	-	-	-	-	-	24	ANNULAR CONCRETE INSERT W/0% B_4C	
-	-	-	-	-	1	-	-	-	-	-	-	-	-	25	ANNULAR CONCRETE INSERT W/1% B_4C	
-	-	-	-	1	-	-	-	-	-	-	-	-	-	26	ANNULAR CONCRETE INSERT W/3% B_4C	
-	-	-	1	-	-	-	-	-	-	-	-	-	-	27	ANNULAR CONCRETE INSERT W/10% B_4C	
-	-	1	-	-	-	-	-	-	-	-	-	-	-	28	SOLID PLASTIC & Cd INSERT	
-	1	-	-	-	-	-	-	-	-	-	-	-	-	29	ANNULAR PLASTIC & Cd INSERT	SH 5
SEE NOTE 13														30	BOTTLE	SH 6
SEE NOTE 13														31	BOTTLE CAP	
1	-	-	-	-	-	-	-	-	-	-	-	-	-	32	SPIDER	
SEE NOTE 13														33	STAND	
SEE NOTE 13														34	CLIP	SH 6
SEE NOTE 13														35	WINDOW 3½" DIA x ½" THK	LEXAN
SEE NOTE 13														36	GASKET 3½" OD x 3" ID x ⅛" THK	NEOPRENE
-	-	-	-	-	-	-	-	-	-	-	AR	AR	AR	37	LAMINATED SHIM STOCK	300 SERIES SST
SEE NOTE 13														38	O-RING #2-235 VITON A	PARKER SEALS
-	-	-	-	-	-	-	-	-	-	-	18	18	15	39	FILLISTER HD SCR #4-40 UNC 2A x ⅝ L	300 SERIES SST
-	-	-	-	-	-	-	-	-	-	-	18	18	15	40	LOCK NUT #4-40 UNC 2B	300 SERIES SST
SEE NOTE 13														41	NUT ¼-20 UNC 2B	300 SERIES SST
SEE NOTE 13														42	½" HEX HD PIPE PLUG	300 SERIES SST
SEE NOTE 15													2	43	RE-ENTRANT TUBE HOLDER	SH 1
SEE NOTE 15													2	44	SWAGELOK 1 ¼" MALE CONNECTOR WITH NYLON FERRULES	#SS-2000-1-20BT
SEE NOTE 15													2	45	GASKET 5" OD x 3" ID x ⅛" THK PUNCH TO MATCH PART 43	NEOPRENE

FORMERLY SK-2-3872

JOP# 1995

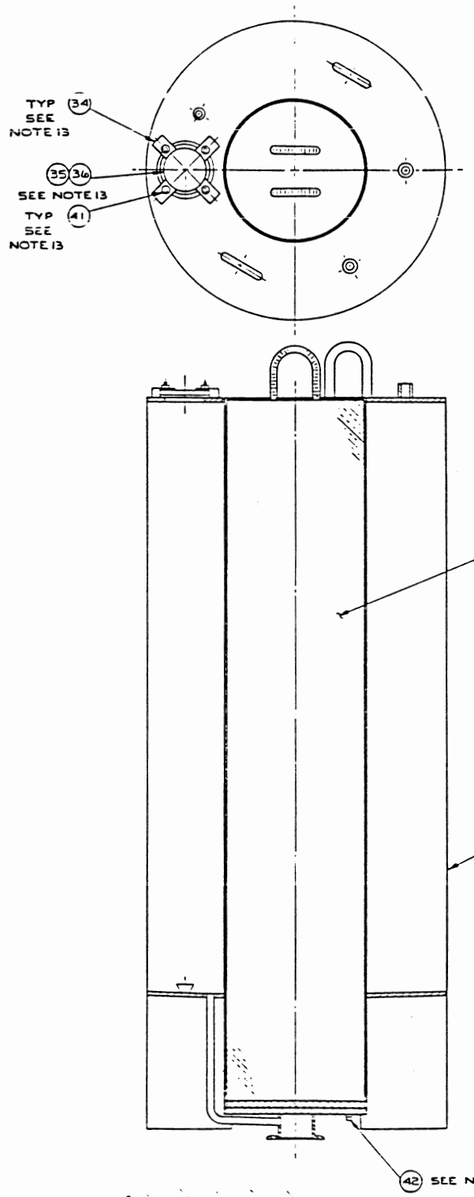
U. S. Department Of Energy
Richland Operations Office
PACIFIC NORTHWEST LABORATORY
OPERATED BY BATTELLE MEMORIAL INSTITUTE

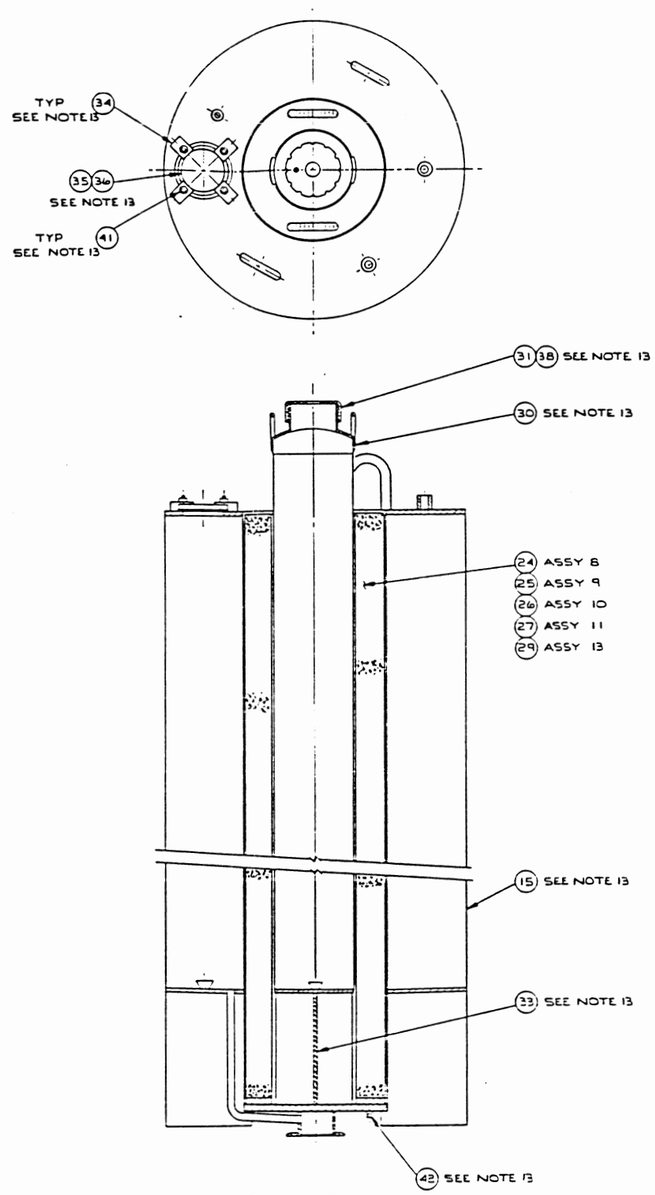
ANNULAR TANK WITH FTR PIN LATTICES

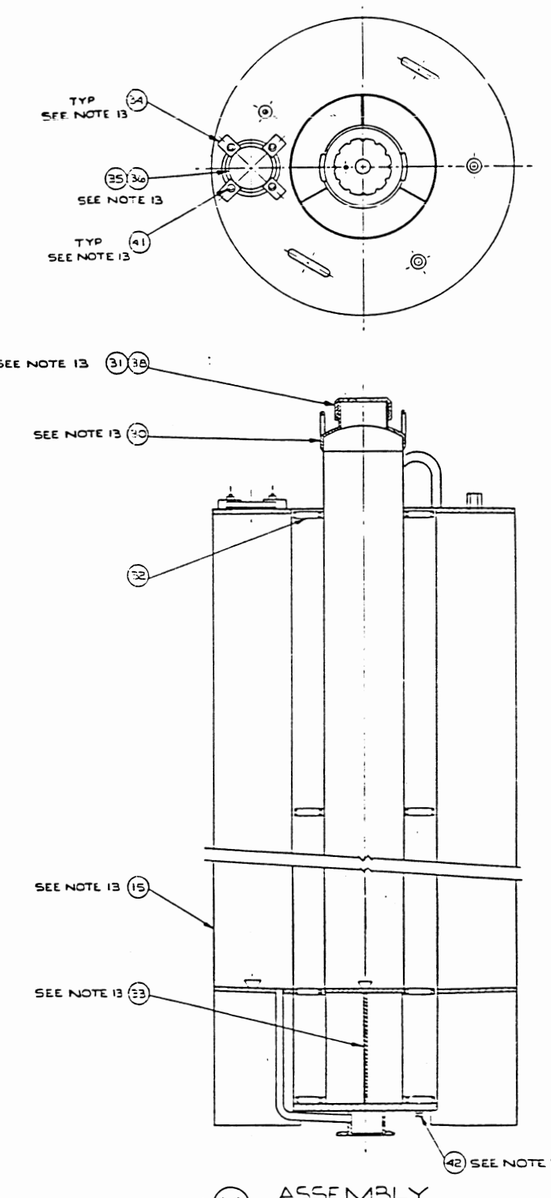
R. L. LLOYD
M. COVERT
NONE
NOT REQD
NONE

20RE
2303
H-2-95741
1 | 6

REVISIONS
REFERENCE DRAWINGS
NEXT USED ON







(16) (4) ASSEMBLY SHOWN

(5) ASSEMBLY AS NOTED

(6) ASSEMBLY AS NOTED

(7) ASSEMBLY AS NOTED

(12) ASSEMBLY SHOWN (16)

(8) ASSEMBLY SHOWN

(9) ASSEMBLY AS NOTED

(10) ASSEMBLY AS NOTED

(11) ASSEMBLY AS NOTED

(13) ASSEMBLY AS NOTED

(14) ASSEMBLY

FORMERLY SK-2-3872
ISSUED 11-26-84

SEE SH 1 FOR GEN NOTES & PARTS LIST

JOB # 7558

U. S. Department Of Energy
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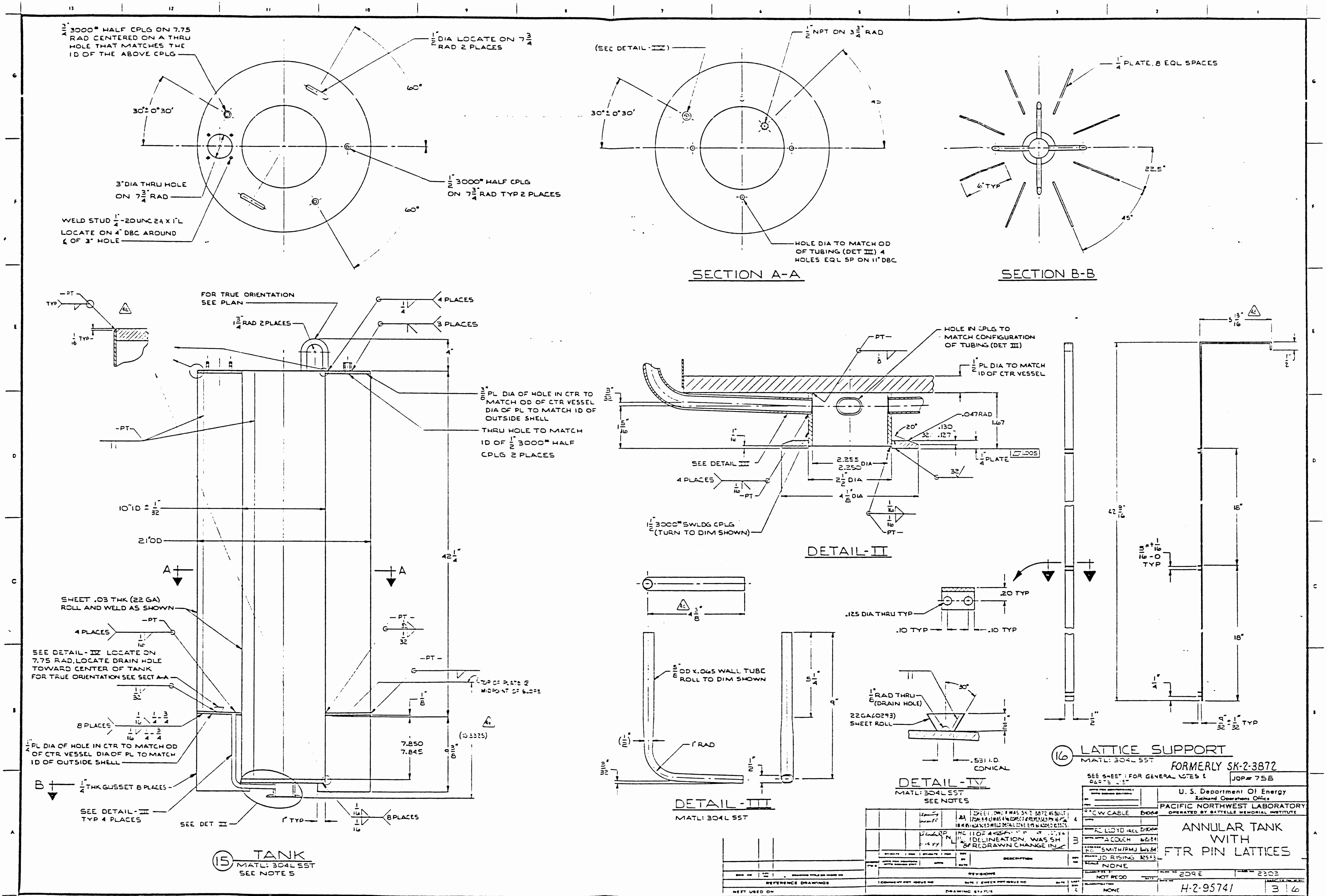
ANNULAR TANK WITH FTR PIN LATTICES

R C LLOYD 11-84
M COVERT 11-84
SCALE NONE
QUALITY NOT REQD
209E
2303
NONE
H-2-95741
2 16

REFERENCE DRAWINGS
NEXT USED ON
REVISIONS
DRAWING STATUS



SECTION A-A
SECTION B-B
DETAIL-II
DETAIL-III
MATL: 304L SST
DETAIL-IV
MATL: 304L SST
SEE NOTES
15 TANK
MATL: 304L SST
SEE NOTE 5
16 LATTICE SUPPORT
MATL: 304L SST
FORMERLY SK-2-3872
JOP# 758
SHEET .03 THK (22 GA) ROLL AND WELD AS SHOWN
FOR TRUE ORIENTATION SEE PLAN
10" ID ± 1/32
21" OD
1/2" NPT ON 3 3/4" RAD
1/4" PLATE, 8 EQL SPACES
HOLE DIA TO MATCH OD OF TUBING (DET III) 4 HOLES EQL SP ON 11" DBC
3" DIA THRU HOLE ON 7 3/4" RAD
WELD STUD 1/4"-20UNC 2A X 1"L LOCATE ON 4" DBC AROUND ℄ OF 3" HOLE
1/2" 3000# HALF CPLG ON 7 3/4" RAD TYP 2 PLACES
1/2" DIA LOCATE ON 7 3/4 RAD 2 PLACES
HOLE IN CPLG TO MATCH CONFIGURATION OF TUBING (DET III)
5/8" OD X .065 WALL TUBE ROLL TO DIM SHOWN
1/2" 3000# SWLDG CPLG (TURN TO DIM SHOWN)
1/4" THK GUSSET 8 PLACES
U. S. Department Of Energy
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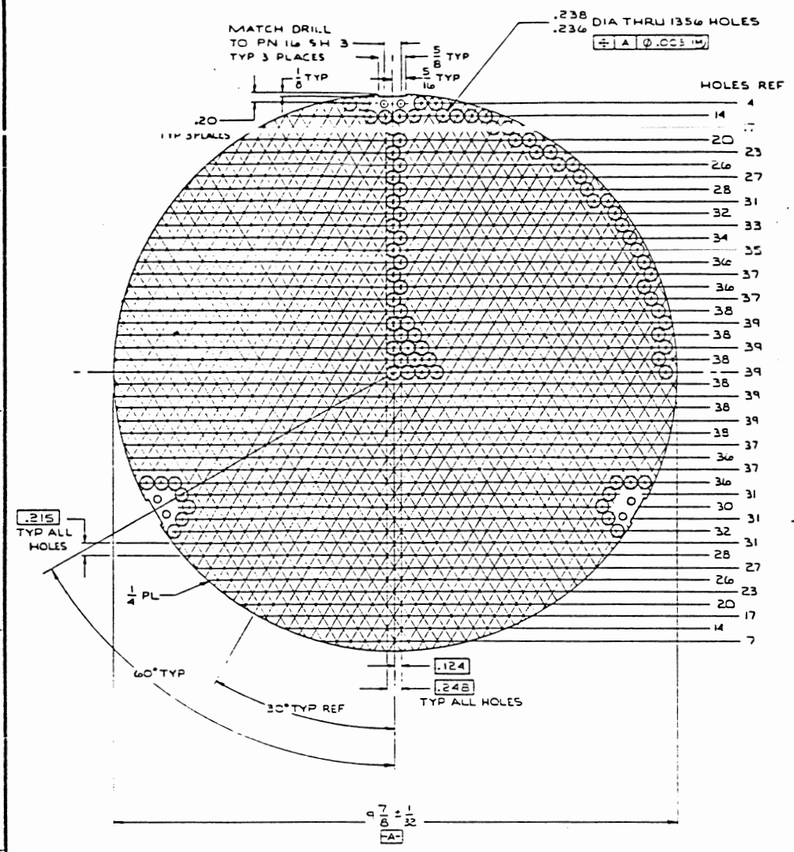
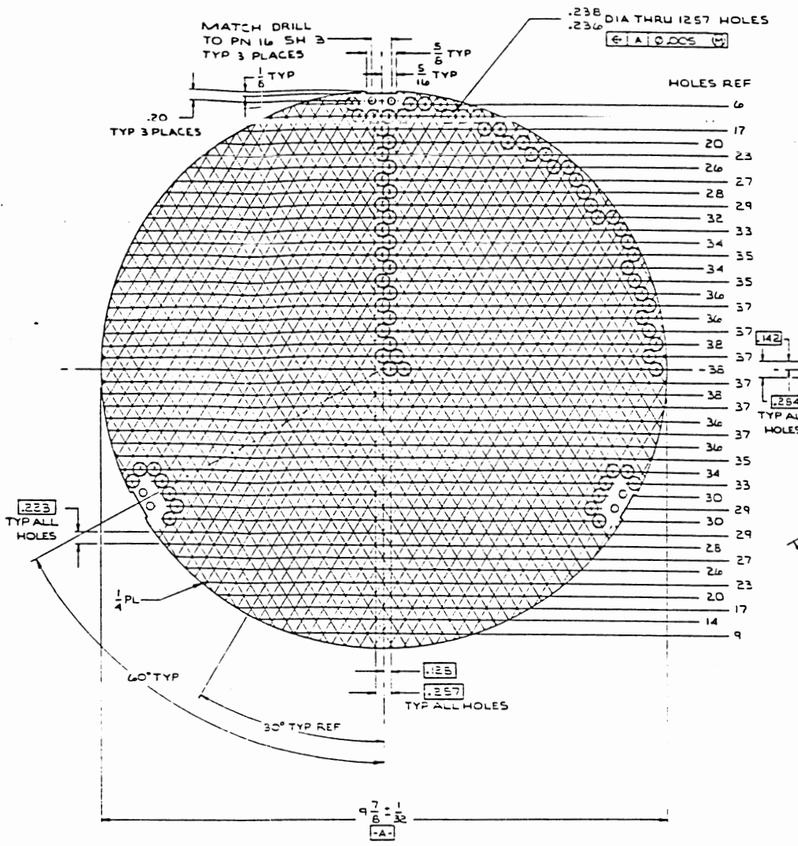
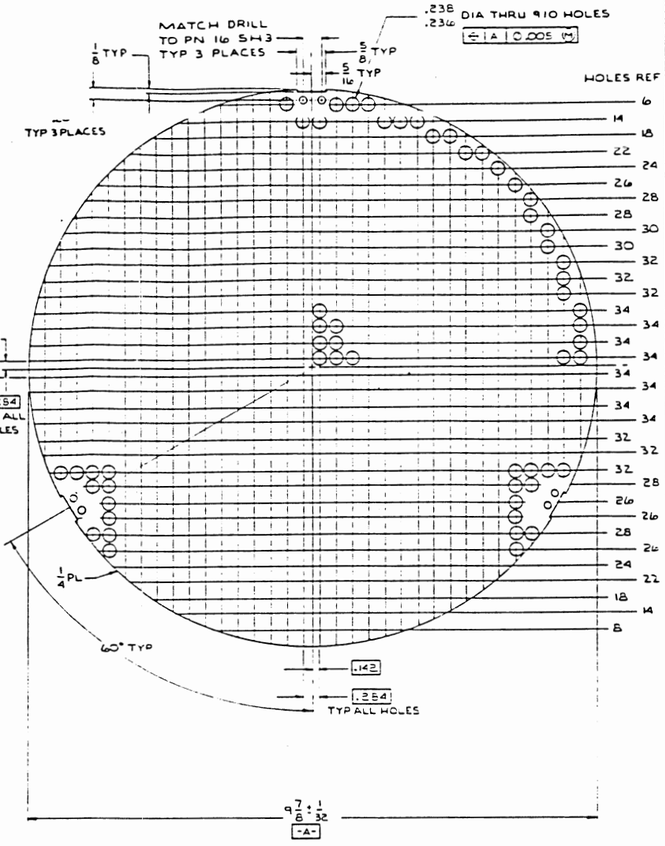
PACIFIC NORTHWEST LABORATORY
OPERATED BY BATTELLE MEMORIAL INSTITUTE
ANNULAR TANK WITH FTR PIN LATTICES
H-2-95741



17 LATTICE PLATE
MATL: LEXAN



18 LATTICE PLATE
MATL: LEXAN



19 LATTICE PLATE
MATL: LEXAN

PARTS LIST & GENERAL NOTES LOCATED ON SH 1

FORMERLY SK-2-3872

ISSUE | 10-28-84 JOB# 758

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PACIFIC NORTHWEST LABORATORY
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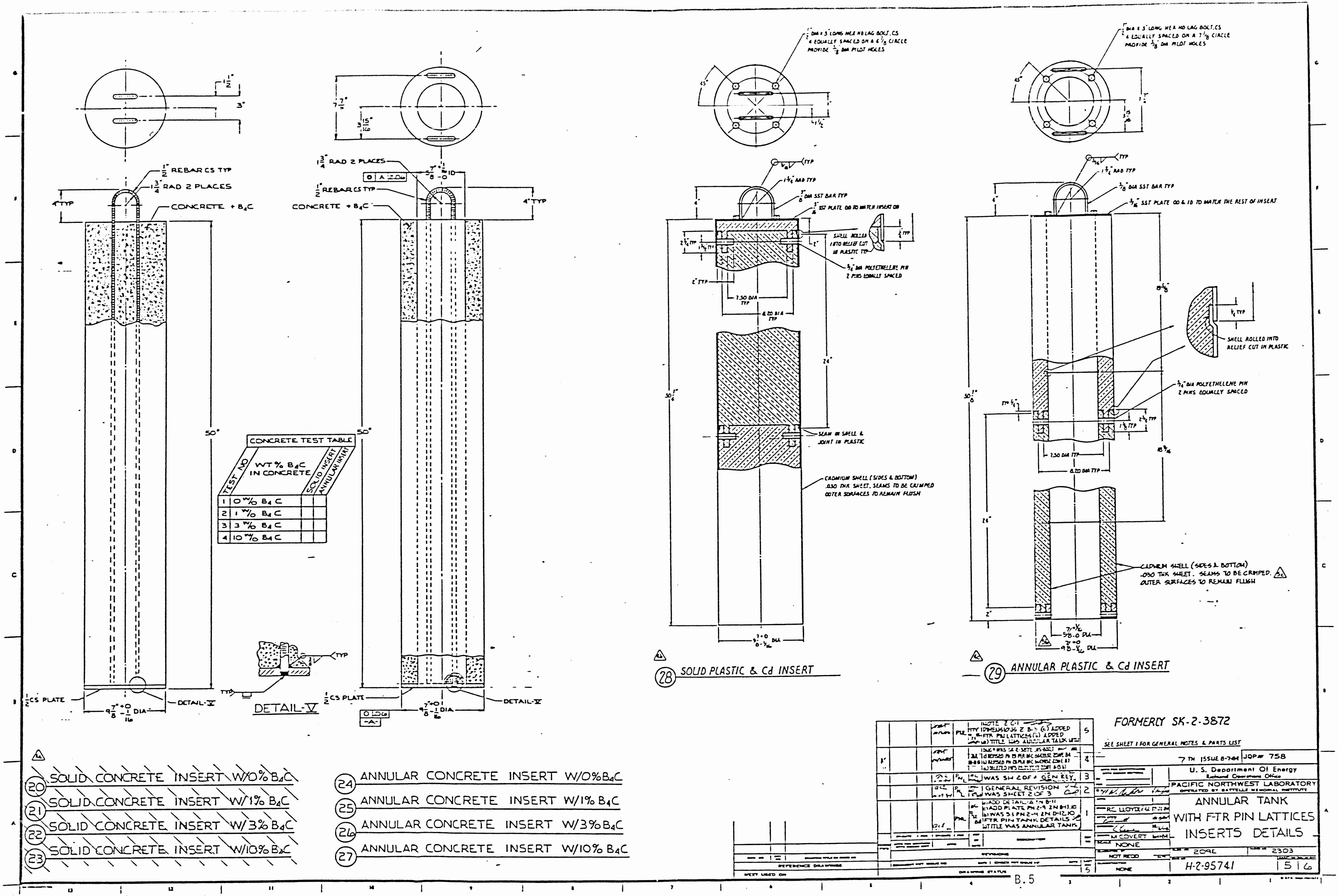
ANNULAR TANK WITH FTR PIN LATTICES DETAILS

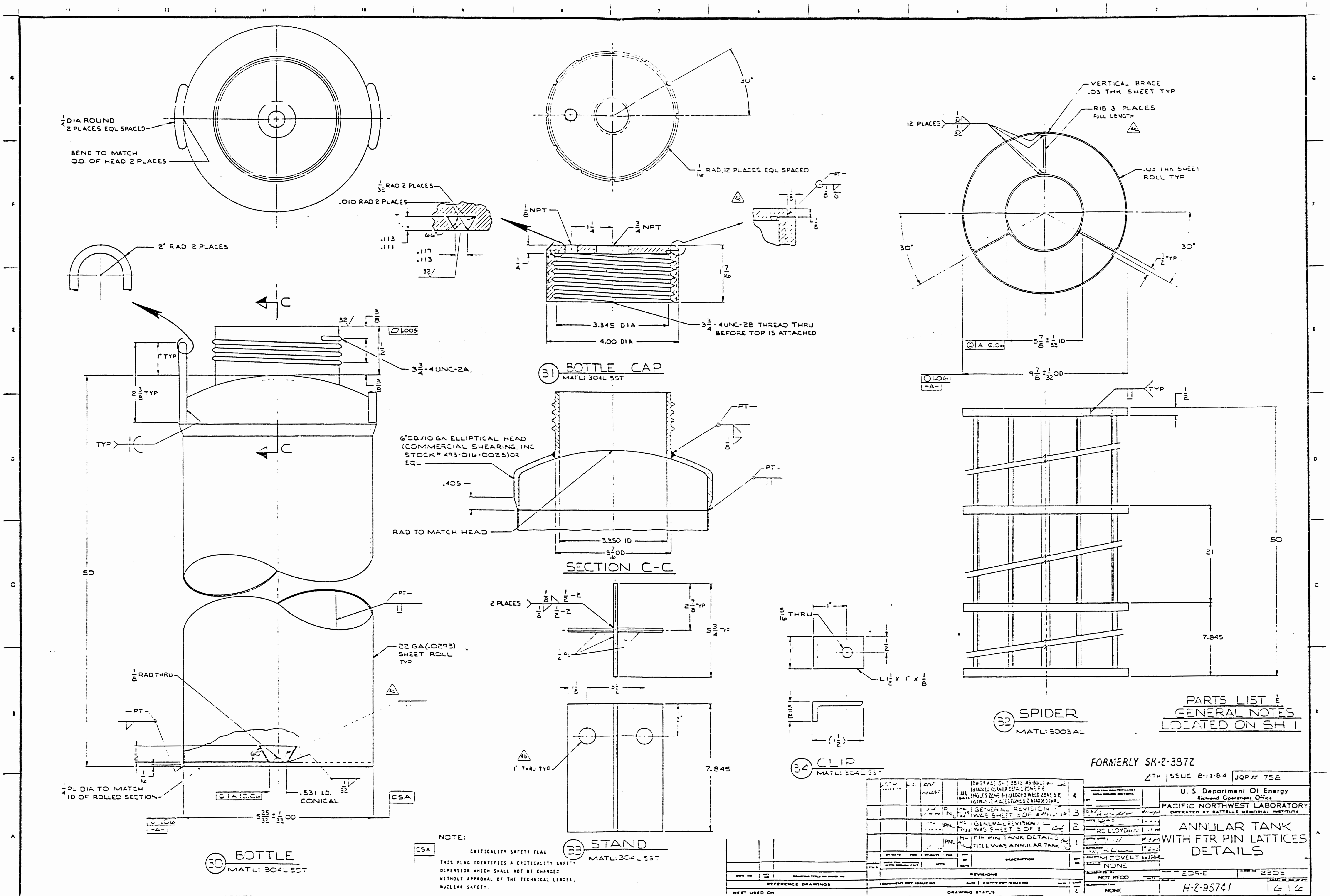
R.C. LLOYD
F.F. CHIARAPPA
M.COVERT
NONE
NOT REQD
NONE
209 E
2303
H-2-95741
4 OF 6

REVISIONS
1 WAS SH 4 OF 4 GEN REV

REFERENCE DRAWINGS
NEXT USED ON
DRAWING STATUS



1/2" REBAR CS TYP
1 3/4" RAD 2 PLACES
CONCRETE + B4C
4" TYP
50"
1/2" CS PLATE
9 7/8 +0 -1/16 DIA
DETAIL-V
CONCRETE TEST TABLE
TEST NO
WT % B4C IN CONCRETE
SOLID INSERT
ANNULAR INSERT
1 0 W/o B4C
2 1 W/o B4C
3 3 W/o B4C
4 10 W/o B4C
SHELL ROLLED INTO RELIEF CUT IN PLASTIC
1/4" DIA POLYETHELENE PIN 2 PINS EQUALLY SPACED
SEAM IN SHELL & JOINT IN PLASTIC
CADMIUM SHELL (SIDES & BOTTOM) .030 THK SHEET. SEAMS TO BE CRIMPED OUTER SURFACES TO REMAIN FLUSH
7.50 DIA TYP
8.20 DIA TYP
24"
28 SOLID PLASTIC & Cd INSERT
29 ANNULAR PLASTIC & Cd INSERT
20 SOLID CONCRETE INSERT W/0% B4C
21 SOLID CONCRETE INSERT W/1% B4C
22 SOLID CONCRETE INSERT W/3% B4C
23 SOLID CONCRETE INSERT W/10% B4C
24 ANNULAR CONCRETE INSERT W/0% B4C
25 ANNULAR CONCRETE INSERT W/1% B4C
26 ANNULAR CONCRETE INSERT W/3% B4C
27 ANNULAR CONCRETE INSERT W/10% B4C
FORMERLY SK-2-3872
SEE SHEET 1 FOR GENERAL NOTES & PARTS LIST
7TH ISSUE 8-7-84
JOP# 758
U. S. Department Of Energy
Richland Operations Office
PACIFIC NORTHWEST LABORATORY
OPERATED BY BATTELLE MEMORIAL INSTITUTE
ANNULAR TANK
WITH F-TR PIN LATTICES
INSERTS DETAILS
RC LLOYD
M COVERT
NONE
NOT REQD
209E
2303
H-2-95741
5 6



1/4 DIA ROUND 2 PLACES EQL SPACED
BEND TO MATCH O.D. OF HEAD 2 PLACES
2" RAD 2 PLACES
C
1" TYP
2 3/8 TYP
TYP
3 3/4 - 4UNC-2A
50
22 GA(.0293) SHEET ROLL TYP
1/8 RAD.THRU
PT
3/4 PL DIA TO MATCH ID OF ROLLED SECTION
.531 I.D. CONICAL
5 25/32 ± 1/32 OD
CSA
30 BOTTLE
MATL: 304L SST
1/32 RAD 2 PLACES
.010 RAD 2 PLACES
.113
.111
.117
.113
1/16 RAD.12 PLACES EQL SPACED
30°
1/8 NPT
3/4 NPT
1 7/16
3.345 DIA
4.00 DIA
3 3/4 - 4UNC-2B THREAD THRU BEFORE TOP IS ATTACHED
31 BOTTLE CAP
MATL: 304L SST
6"OD.X10 GA ELLIPTICAL HEAD (COMMERCIAL SHEARING, INC STOCK# 493-016-0025)OR EQL
.405
RAD TO MATCH HEAD
3.250 ID
3 7/16 OD
SECTION C-C
2 PLACES
5 3/4 TYP
7.845
33 STAND
MATL: 304L SST
1" THRU TYP
5/16 THRU
L 1 1/2 x 1" x 1/8
34 CLIP
MATL: 304L SST
VERTICAL BRACE .03 THK SHEET TYP
RIB 3 PLACES FULL LENGTH
12 PLACES
.03 THK SHEET ROLL TYP
5 7/8 ± 1/32 ID
9 7/8 ± 1/32 OD
21
50
7.845
32 SPIDER
MATL: 5003 AL
PARTS LIST & GENERAL NOTES LOCATED ON SH 1
NOTE:
CRITICALITY SAFETY FLAG
THIS FLAG IDENTIFIES A CRITICALITY SAFETY DIMENSION WHICH SHALL NOT BE CHANGED WITHOUT APPROVAL OF THE TECHNICAL LEADER, NUCLEAR SAFETY.
FORMERLY SK-2-3372
4TH ISSUE 8-13-84
JQP # 758
DWG WAS SK-2-3372 AS-BUILT
GENERAL REVISION WAS SHEET 3 OF 4
GENERAL REVISION WAS SHEET 3 OF 3
FTR PIN TANK DETAILS TITLE WAS ANNULAR TANK
REVISIONS
REFERENCE DRAWINGS
DRAWING STATUS
RC LLOYD
M COVERT
NONE
NOT REQD
U. S. Department Of Energy
Richland Operations Office
PACIFIC NORTHWEST LABORATORY
OPERATED BY BATTELLE MEMORIAL INSTITUTE
ANNULAR TANK WITH FTR PIN LATTICES DETAILS
209-E
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H-2-95741
6 | 6

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 annular tank. The solution height values used in these plots are given in inches.

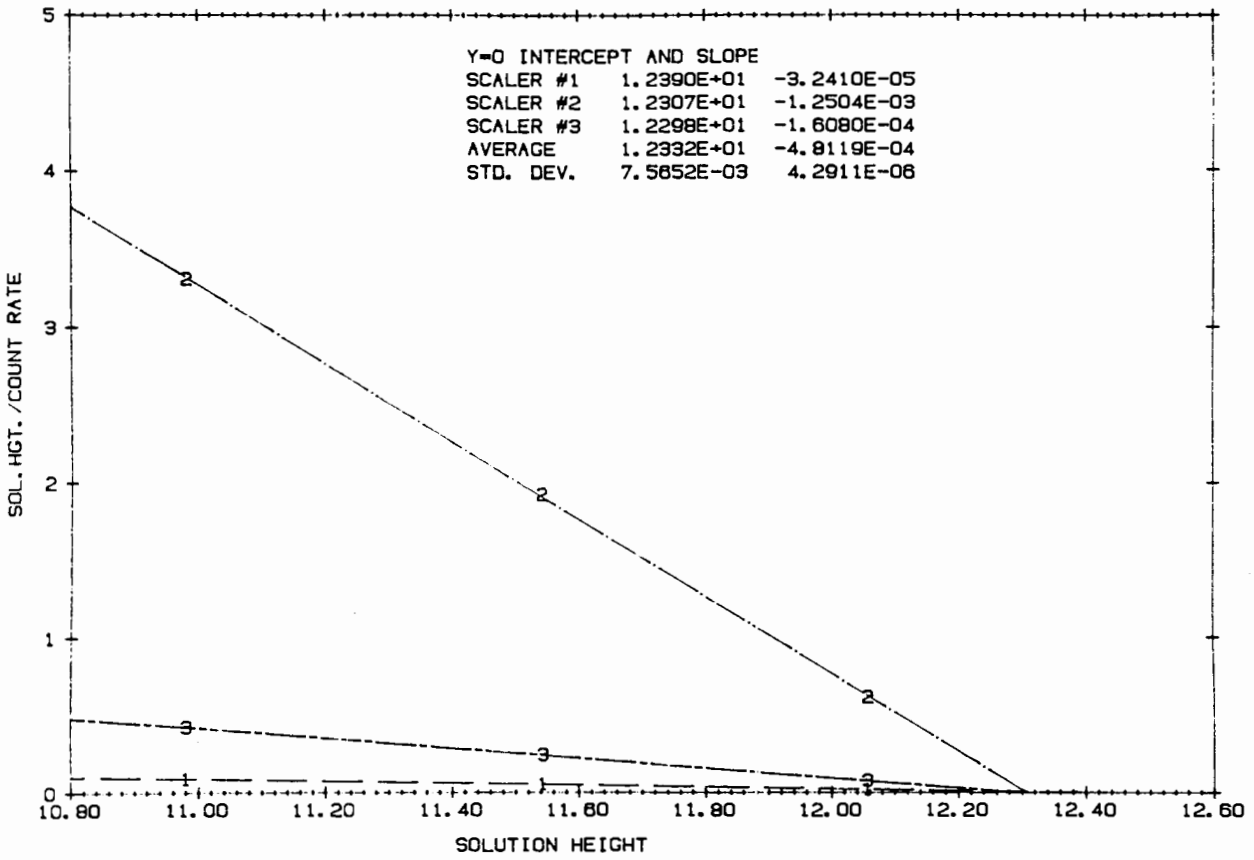

FIGURE C.1 Least Square Fits for CFRP-PNC 052

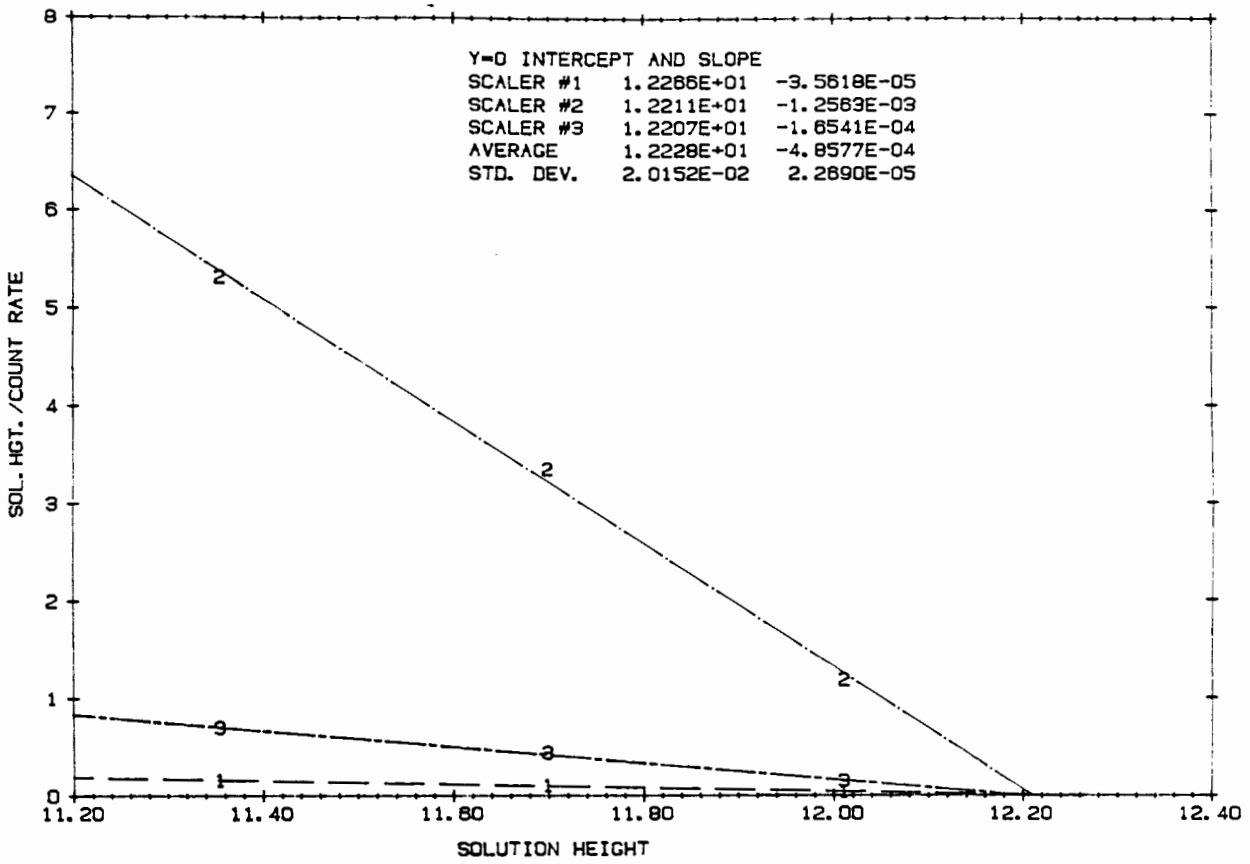

FIGURE C.2 Least Square Fits for CFRP-PNC 052R

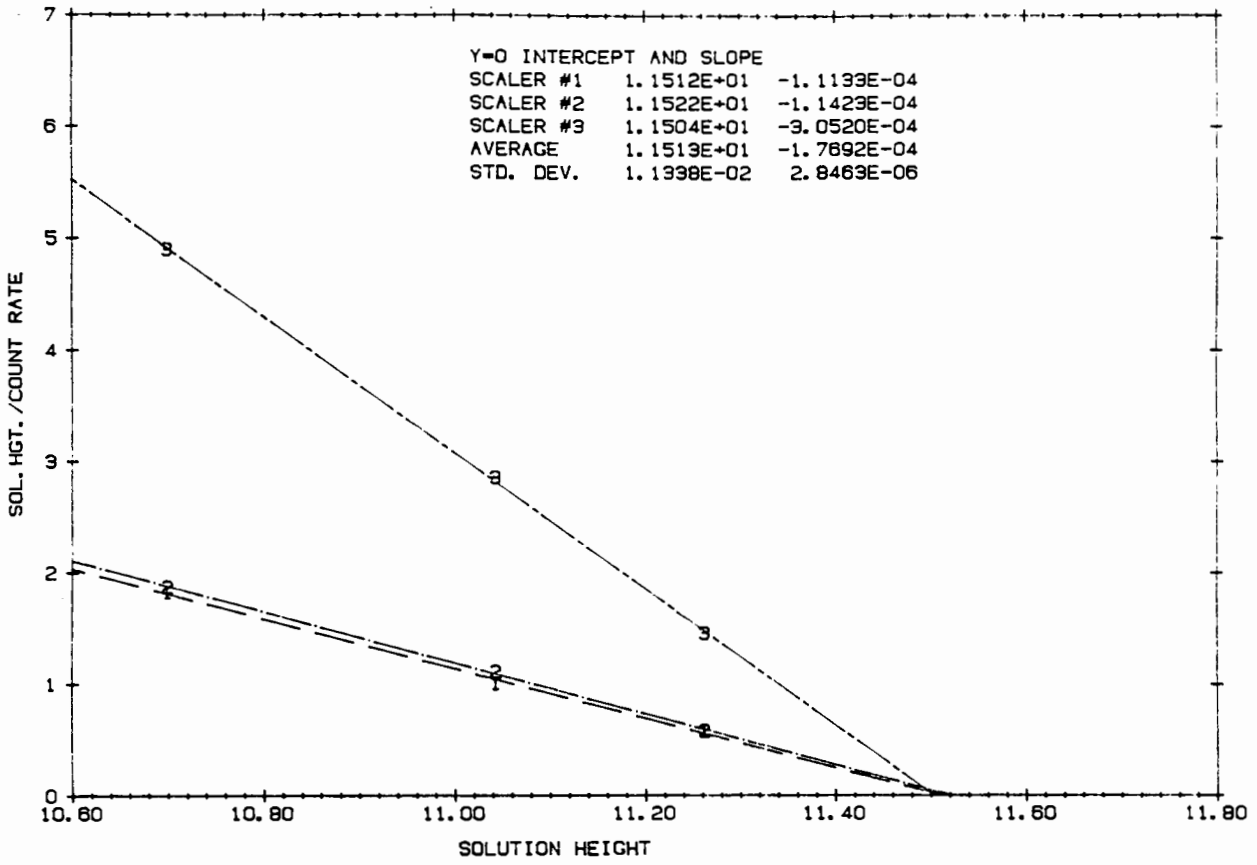

FIGURE C.3 Least Square Fits for CFRP-PNC 053

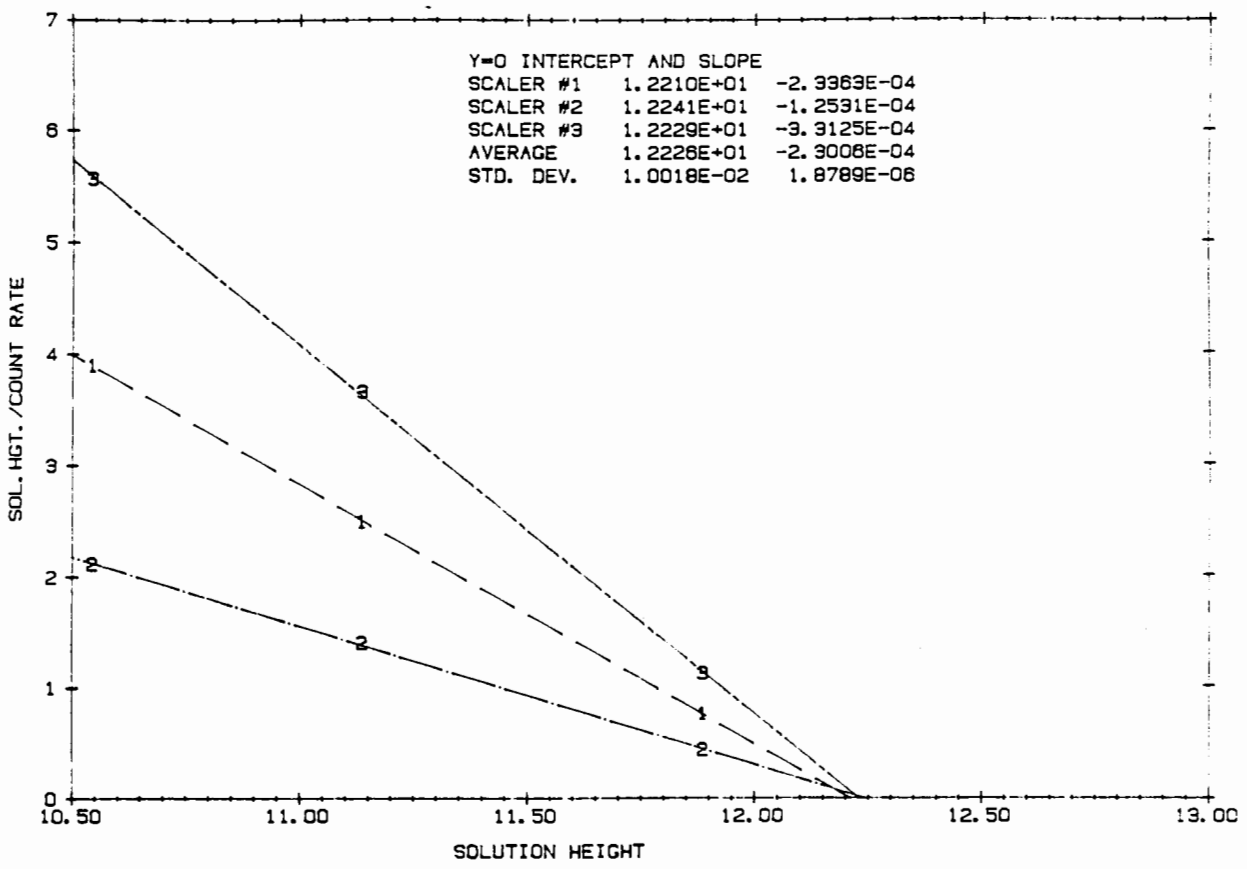

FIGURE C.4 Least Square Fits for CFRP-PNC 057

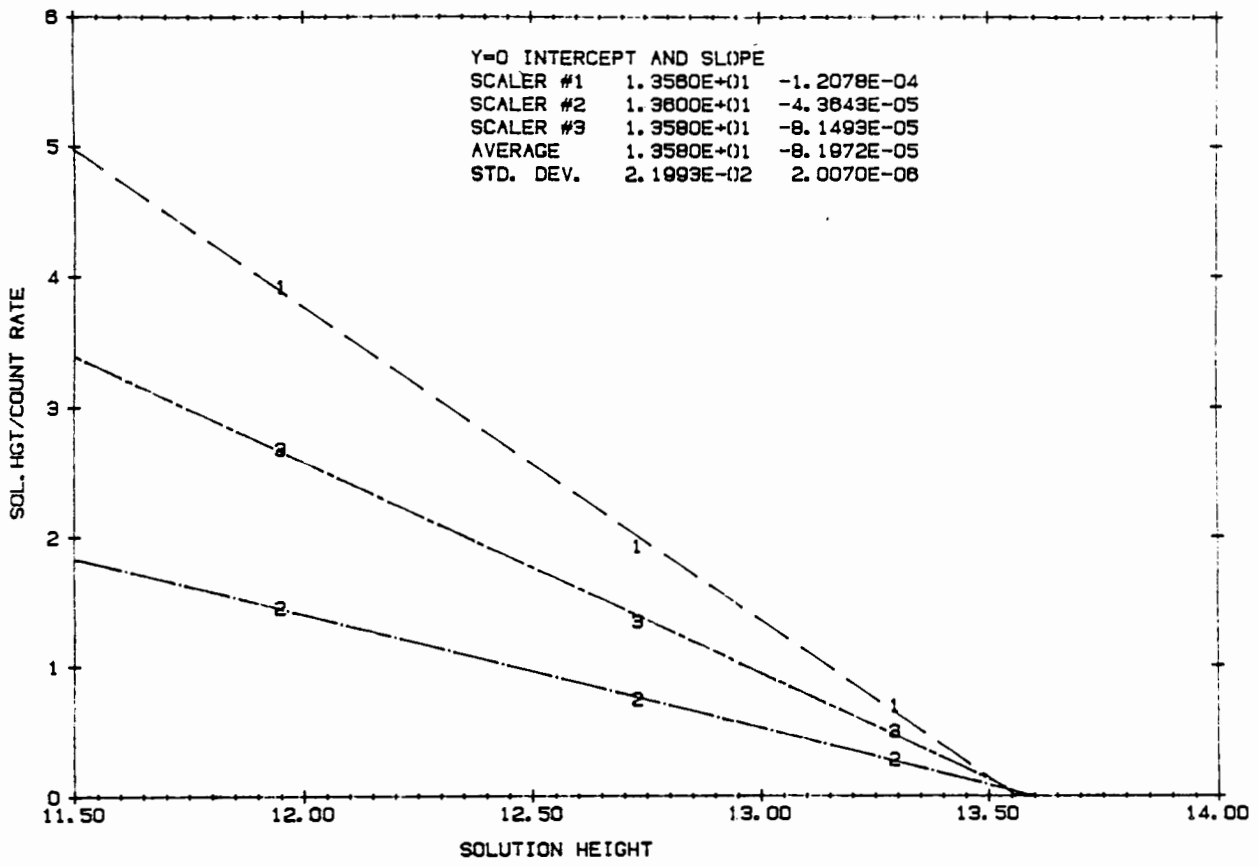

FIGURE C.5 Least Square Fits for CFRP-PNC 062

APPENDIX D

ANALYSIS OF PLUTONIUM CONCENTRATION DATA

APPENDIX D

ANALYSIS OF PLUTONIUM CONCENTRATION DATA

Initially, the chemical analysis procedure used to determine the plutonium concentration of critical experiment samples did not involve a step where the solution is ion exchanged. The ion exchange procedure separates the plutonium from impurities in solution. Since the impurities were left in the solution they interfered with the analysis so that the indicated plutonium concentration contained a positive bias proportional to the concentration of impurities. This procedure was used on some samples in this report and for samples in the series of critical experiments reported in PNL-5768 (Lloyd 1986).

In order to determine the magnitude of the positive bias and correct the plutonium data in the reports, archive critical experiment solution samples were reanalyzed in December 1987. The analyses were performed both with and without ion exchanging the solutions since it was expected that concentration of the solutions may have changed over the course of two years. The results of these analyses along with the original analysis results are shown in Table D.1. Comparing the results from the original analysis to the recent analyses (both without ion exchange), it was found that the solutions had concentrated by an average of 1.4 ± 0.3 percent. This conclusion is also supported by the comparison of the density measurement results. Because the solutions were stored for up to two years in polyethylene bottles the concentration of solutions is not surprising. As a result of the apparent evaporation, the recent plutonium analysis results, which include ion exchange in the procedure, could not be used directly. Instead, the ratio of the non-ion exchanged results to the ion exchanged results was used to correct the original analyses. The ratio was found to be 1.006 ± 0.002. The ratio was used to obtain corrected plutonium concentrations as shown in the last column of Table D.1. These corrected values were used in Table 3.1 of this report and in Tables 3.1 and 3.2 of PNL-5768.

TABLE D.1 Chemical Analysis Results

Sample Number	Experiment Number	PNL Report Number	Original Analysis Date	Original Pu (g/liter)	Original Density (g/cm^3)	NEW-NIX[a] Pu (g/liter)	NEW-IX[b] Pu (g/liter)	New Density (g/cm^3)	Corrected Pu (g/liter)
1087	046, 046R	5768	1-85	59.35	1.1968	60.36	59.88	1.1988	59.0
1088	047	5768	2-85	59.82	1.1977	60.74	60.42	1.2005	59.5
1095	048, 49A, 050	5768	5-85	174.07	1.5316	176.46	175.38	1.5370	173.0
1096	051	5768	5-85	59.63	1.1978	60.60	60.50	1.2157	59.3
1111	052, 052R	6119	9-85	173.66	1.5295	175.56	174.38	1.5342	172.6
1112	053	6119	9-85	113.71	1.3569	115.20	114.55	1.3641	113.0
1119	054	5768	9-85	118.94	1.3714	120.03	119.18	1.3742	118.2
1120	055	5768	9-85	60.53	1.1944	61.58	61.18	1.2042	60.2
1121	056, 056a	5768	9-85	61.00	1.1954	61.77	61.45	1.1982	60.6
1122	057	6119	9-85	61.02	1.1957	61.73	61.38	1.1977	60.7

(a) NEW-NIX means analysis performed without ion exchange.

(b) NEW-IX means analysis performed with ion exchange.

APPENDIX E

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

APPENDIX E

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

The chemical analyses data from sample 1110 is for the solution in the bottle insert.

The chemical analyses data from sample 1111 is for experiments 052 and 052R. The chemical analyses data from sample 1112 is for experiment 053. The chemical analyses data from sample 1121 is for experiment 057. The chemical analyses data from sample 1152 is for experiment 062.

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 E.1 Spectrographic Analysis Report # 1110

Hanford Engineering Development Laboratory

SPECTROCHEMICAL ANALYSIS REPORT

CC: SPEC. LAB.

SC 100, 101

MATERIAL $PuO_2 - UO_2$

SUBMITTED BY	SUBMITTER'S NO.	ANALYZED BY	DATE REPORTED
E. MURPHY	1110 A	RK	9/6/83

ELEMENT			ELEMENT			ELEMENT			ELEMENT	
Ag	<10	<10	Hg			Re			Dy	
Al	500	1000	In			Rh			Er	
As			Ir			Ru			Eu	
Au			K	200	300	Sb			Gd	
B	6	7	La			Sc			Ho	
Ba			Li	<1	<1	Si	<40	<40	Lu	
Be	<2	<2	Mg	100	>200	Sn	<10	<10	Nd	
Bi			Mn	30	60	Sr			Pr	
Ca	<25	200	Mo	<30	<30	Ta			Sm	
Cd	6	6	Na	200	200	Th			Tb	
Ce			Nb			Ti	<35	300	Tm	
Co	5	6	Ni	<50	<50	Tl			Yb	
Cr	300	200	Os			U				
Ca			P			V	<50	<50		
Cu	200	300	Pb	<10	100	W				
Fe	700	>1000	Pd			Y				
Ga			Pt			Zn	20	<20		
Ge			Pu			Zr				
Hf			Rb							

TYPE OF ANALYSIS

☐ QUALITATIVE

SYMBOL	MEANING	APP'X. CONC
ss	MAJOR DETECTABLE CONSTITUENT	
S	STRONG	GREATER THAN 1 %
M	MODERATE	1 % TO 0.01 %
T	TRACE	LESS THAN 0.01 %
—	NOT DETECTED	
*	INTERFERENCE	
?	DETECTION UNCERTAIN. INTERFERENCE	

☒ SEMIQUANTITATIVE

SYMBOL	MEANING
G	CONCENTRATION GREATER THAN
L	DETECTABLE CONCENTRATION LESS THAN
—	NOT DETECTED

NUMERICAL VALUES: ☒ PARTS PER MILLION ☐ PERCENT ☐

APPR'X. PRECISION ± FACTOR 3

☐ QUANTITATIVE

SYMBOL	MEANING
GX (LX)	CONC. GREATER THAN (LESS THAN) CALIBRATED WORKING CURVE

NUMERICAL VALUES: ☐ PARTS PER MILLION ☐ PERCENT ☐

APPR'X. PRECISION ±

REMARKS:

REPORT APPROVED

LABORATORY INFORMATION

SPECTROGRAPH AND SOURCE	SIZE OF SAMPLE	METHOD OF ANALYSIS	PLATE NO.

BD-7240-021 (8-78)

TABLE E.2 Spectrographic Analysis Report # 1111

SC #119, #120	Hanford Engineering Development Laboratory SPECTROCHEMICAL ANALYSIS REPORT	CC: SPEC. LAB.

MATERIAL: mixed oxide

SUBMITTED BY	SUBMITTER'S NO.	ANALYZED BY	DATE REPORTED
R. LLOYD		JE	10/24/85

ELEMENT	#119	#120	ELEMENT	#119	#120	ELEMENT	#119	#120	ELEMENT
Ag	< 10	< 10	Hg			Re			Dy
Al	370	700	In			Rh			Er
As			Ir			Ru			Eu
Au			K	140	230	Sb			Gd
B	8	10	La			Sc			Ho
Ba			Li	< 1	< 1	Si	< 40	< 40	Lu
Be	< 2	< 2	Mg	20	40	Sn	< 10	< 10	Nd
Bi			Mn	60	80	Sr			Pr
Ca	100	250	Mo	< 30	40	Ta			Sm
Cd	3	5	Na	< 60	< 60	Th			Tb
Ce			Nb			Ti	< 35	< 35	Tm
Co	7	7	Ni	270	320	Tl			Yb
Cr	340	400	Os			U			
Cs			P			V	< 50	< 50	
Cu	< 20	< 20	Pb	< 10	< 10	W			
Fe	1300	1600	Pd			Y			
Ga			Pt			Zn	20	40	
Ge			Pu			Zr			
Hf			Rb						

TYPE OF ANALYSIS

☐ QUALITATIVE

SYMBOL	MEANING	APP'X. CONC.
SS	MAJOR DETECTABLE CONSTITUENT	
S	STRONG	GREATER THAN 1 %
M	MODERATE	1 % TO 0.01 %
T	TRACE	LESS THAN 0.01 %
—	NOT DETECTED	
*	INTERFERENCE	
?	DETECTION UNCERTAIN. INTERFERENCE	

☑ SEMIQUANTITATIVE

SYMBOL	MEANING
G	CONCENTRATION GREATER THAN
L	DETECTABLE CONCENTRATION LESS THAN
—	NOT DETECTED

NUMERICAL VALUES: ☑ PARTS PER MILLION ☐ PERCENT ☐ _____

APPR'X. PRECISION ± FACTOR 2

☐ QUANTITATIVE

SYMBOL	MEANING
GX (LX)	CONC. GREATER THAN (LESS THAN) CALIBRATED WORKING CURVE

NUMERICAL VALUES: ☐ PARTS PER MILLION ☐ PERCENT ☐ _____

APPR'X. PRECISION ± _____

REMARKS:

#119 = 1095 A & B

#120 = 1111 A

REPORT APPROVED Roy Ko

LABORATORY INFORMATION

SPECTROGRAPH AND SOURCE	SIZE OF SAMPLE	METHOD OF ANALYSIS	PLATE NO.

BD-7340-021 (3-73)

TABLE E.3 Spectrographic Analysis Report # 1112

SC 102, 103

Hanford Engineering Development Laboratory

SPECTROCHEMICAL ANALYSIS REPORT

CC: SPEC. LAB.

MATERIAL: PuO_2-UO_2

SUBMITTED BY	SUBMITTER'S NO	ANALYZED BY	DATE REPORTED
E. MURPHY	1112-A	RK	9/6/85

ELEMENT			ELEMENT			ELEMENT			ELEMENT	
Ag	<10	<10	Hg			Re			Dy	
Al	500	500	In			Rh			Er	
As			Ir			Ru			Eu	
Au			K	600	400	Sb			Gd	
B	10	10	La			Sc			Ho	
Ba			Li	<1	<1	Si	<40	<40	Lu	
Be	<2	<2	Mg	100	100	Sn	<10	<10	Nd	
Bi			Mn	70	80	Sr			Pr	
Ca	<25	<25	Mo	<30	<30	Ta			Sm	
Cd	10	10	Na	500	400	Th			Tb	
Ce			Nb			Ti	<35	<35	Tm	
Co	6	6	Ni	<50	<50	Tl			Yb	
Cr	600	700	Os			U				
Cs			P			V	<50	<50		
Cu	500	600	Pb	>200	>200	W				
Fe	>1000	>1000	Pd			Y				
Ga			Pt			Zn	60	200		
Ge			Pu			Zr				
Hf			Rb							

TYPE OF ANALYSIS

☐ QUALITATIVE

SYMBOL	MEANING	APP'X CONC
SS	MAJOR DETECTABLE CONSTITUENT	
S	STRONG	GREATER THAN 1 %
M	MODERATE	1 % TO 0.01 %
T	TRACE	LESS THAN 0.01 %
—	NOT DETECTED	
*	INTERFERENCE	
?	DETECTION UNCERTAIN. INTERFERENCE	

☒ SEMIQUANTITATIVE

SYMBOL	MEANING
G	CONCENTRATION GREATER THAN
L	DETECTABLE CONCENTRATION LESS THAN
—	NOT DETECTED

NUMERICAL VALUES: ☒ PARTS PER MILLION ☐ PERCENT ☐

APPR'X. PRECISION ± FACTOR 2

☐ QUANTITATIVE

SYMBOL	MEANING
SX (LX)	CONC. GREATER THAN (LESS THAN) CALIBRATED WORKING CURVE

NUMERICAL VALUES: ☐ PARTS PER MILLION ☐ PERCENT ☐ _____

APPR'X. PRECISION ±

REMARKS:

REPORT APPROVED

LABORATORY INFORMATION

SPECTROGRAPH AND SOURCE	SIZE OF SAMPLE	METHOD OF ANALYSIS	PLATE NO.

HO-7340-021 (3-73)

TABLE E.4 Spectrographic Analysis Report # 1121

LOG NO. 123, 125	Hanford Engineering Development Laboratory	SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT

MATERIAL: WASTE SOLUTIONS

SUBMITTED BY	SUBMITTER'S NO.	ANALYZED BY	DATE REPORTED
E. S. MURPHY	SEE BELOW	RK	9/26/81

ELEMENT	123	125	ELEMENT	123	125	ELEMENT	
Li	0.2	0.2	Ga	30	30	Pm	
Be	*	*	Ge			Sm	
B	100	100	As			Eu	
F			Rb			Gd	
Na	1000	1000	Sr			Tb	
Mg	300	300	Y			Dy	
Al	2000	2000	Zr			Ho	
Si	2000	2000	Nb			Er	
P	30	30	Mo	2	2	Tm	
S	200	300	Ru			Yb	
Cl	10	100	Rh			Lu	
K	100	300	Pd			Hf	
Ca	5	50	Ag	*	*	Ta	
Sc			Cd	1	1	W	
Ti	40	40	In			Re	
V	0.1	<0.3	Sn			Os	
Cr	30	50	Sb			Ir	
Mn	7	7	Cs			Pt	
Fe	30	90	Ba	4	4	Au	
Co	0.2	0.2	La			Hg	
Ni	30	30	Ce			TH	✓ ✓
Cu	200	600	Pr			Pb	
Zn	40	40	Nd			Bi	

TYPE OF ANALYSIS

☐ QUALITATIVE	☒ SEMIQUANTITATIVE	☐ QUANTITATIVE
☐ PARTS PER MILLION	☒ PARTS PER MILLION) µg/g	☐ PARTS PER MILLION
☐ PERCENT	☐ PERCENT	☐ PERCENT
☐ _____	☐ _____	☐ _____
APPR'X PRECISION ± FACTOR _____	APPR'X PRECISION ± FACTOR 3	APPROX PRECISION ± FACTOR _____

REMARKS:
SOLUTIONS WERE EVAPORATED & IGNITED AT 700C. RESULTS ARE IN µg/g of IGNITED RESIDUE
123 = 1119 A
125 = 1121 A

* INTERFERENCE

REPORT APPROVED RK

BD-7340-021 (4-77)

TABLE E.5 Spectrographic Analysis Report # 1152

LOG NO. #33	Hanford Engineering Development Laboratory	SPARK SOURCE MASS SPECTROGRAPHIC ANALYSIS REPORT

MATERIAL: mixed oxide

SUBMITTED BY	SUBMITTER'S NO.	ANALYZED BY	DATE REPORTED
E.S. Murphy	1152-C	JE	5/15/86

ELEMENT	#33	ELEMENT		ELEMENT	
Li	1	Ga		Pm	
Be	0.4	Ge		Sm	1
B	3	As		Eu	1
F		Rb		Gd	2
Na	30	Sr		Tb	
Mg	7	Y		Dy	2
Al	30	Zr	40	Ho	
Si	10	Nb		Er	
P	10	Mo	6	Tm	
S		Ru		Yb	
Cl		Rh		Lu	
K	4	Pd		Hf	
Ca	200	Ag		Ta	
Sc		Cd	1	W	2
Ti	40	In		Re	
V	0.3	Sn	2	Os	
Cr	200	Sb		Ir	
Mn	70	Cs		Pt	
Fe	300	Ba		Au	
Co	3	La		Hg	
Ni	100	Ce		Tl	
Cu	400	Pr		Pb	
Zn	100	Nd		Bi	

TYPE OF ANALYSIS

QUALITATIVE	SEMIQUANTITATIVE	QUANTITATIVE
☐ PARTS PER MILLION	☑ PARTS PER MILLION	☐ PARTS PER MILLION
☐ PERCENT	☐ PERCENT	☐ PERCENT
☐ _____	☐ _____	☐ _____
APPR'X PRECISION ± FACTOR _____	APPR'X PRECISION ± Factor 3	APPROX PRECISION ± FACTOR _____

REMARKS: 6-3358 Widmer

1152

REPORT APPROVED

BD-7340-021 (4-77)

TABLE E.6 Inductively Coupled Plasma Spectroscopy Analysis (mg/liter)

Element	Sample Number 1121
Al	50.1
B	1.4
Ba	3.2
Ca	33.9
Cd	3.5
Ce	1.9
Cr	41.0
Cu	13.8
Dy	0.4
Fe	133.7
Gd	<1
K	8.0
La	0.4
Li	0.3
Mg	8.5
Mn	8.4
Mo	0.6
Na	30.6
Nd	1.3
Ni	30.6
Rh	3.8
Ru	2.1
Si	35.7
Sr	(a)
Te	(a)
Ti	9.0
Zn	2.3
Zr	0.7

(a) Element not detected

APPENDIX F

CHEMICAL ANALYSES DATA OF THE REFLECTOR WATER SAMPLES

TABLE F.1 Water Sample Analyses 052, 053, and 057

HANFORD ENVIRONMENTAL HEALTH FOUNDATION

February 24, 1986 CO 10311

Pacific Northwest Laboratory
209E Building
200E Area

Attn: R. Lloyd

WATER SAMPLE ANALYSES

Results of the analysis of water samples received January 6 and 10, 1986, are following. All analyses were done as specified in Standard Methods for the Analysis of Water and Waste Water, 16th Ed.

Parameter	058	061TR	061TR -2	Tamper 052/053	057
pH	6.9	7.0	7.4	7.6	8.1
Total Alkalinity (mg/L)	45.7	49.2	49.4	44.0	50.9
Bicarbonate Alkalinity (mg/L)	44	47	47	42	48
Carbonate Alkalinity (mg/L)	<0.5	<0.5	<0.5	<0.5	0.7
Total Dissolved Solids (mg/L)	82	86	84	82	81
Sulfate (mg/L)	16.2	16.5	16.4	13.0	15.0
Nitrate (as N) (mg/L)	<0.05	<0.05	0.12	<0.04	0.05
Chloride (mg/L)	2.1	2.1	2.1	3.0	2.4
Fluoride (mg/L)	0.12	0.12	<0.1	<0.1	<0.1
Cadmium (mg/L)	0.0011	<0.0005	<0.0005	0.0008	0.0008
Copper (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Chromium (mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01
Iron (mg/L)	<0.03	<0.03	0.06	0.08	0.11
Lead (mg/L)	<0.002	<0.002	<0.002	<0.002	<0.002
Manganese (mg/L)	<0.01	<0.01	<0.01	0.01	<0.01
Zinc (mg/L)	<0.05	<0.05	0.06	0.18	0.40

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

P. A. Thurman
Environmental Health Sciences

lmk

P. O. BOX 100, RICHLAND, WASHINGTON 99352

TABLE F.2 Water Sample Analysis - 062

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
Environmental Health Sciences

jt

P O BOX 100 RICHLAND WASHINGTON 99352

APPENDIX G

COMPOSITION OF THE CONCRETE INSERTS

APPENDIX G

COMPOSITION OF THE CONCRETE INSERTS

In March 1985, four annular concrete inserts were fabricated for use in critical experiments at the CML in conjunction with an annular cylinder. The four inserts are nearly identical except for varying amount of B_4C. The components used in making each of the inserts are shown in Table G.1. It should be recognized that the fraction of each component in the cured concrete will change since the water content of the concrete will change as the concrete sets. Also, the water content shown only represents the pure water added, and does not include the amount of water intrinsically part of the other components, especially the aggregate.

The Hanford Engineering Development Laboratory performed an isotopic analysis of the boron used in the B_4C. The analysis indicates that ^{10}B constitutes 20.02 ± 0.1 atom percent, with the remainder being ^{11}B. The certificate of analysis from the supplier of the B_4C is shown in Table G.2.

The density of each insert was determined to be 2.19 ± 0.05 g/cc, based on the weight (1.5 Kg) and volume (0.7 liter) of a sample of one of the concrete inserts. This value was confirmed by analysis of the total weight and volume of each insert. Also the concrete inserts were weighed at various times between October 1985 and January 1988 and the weights did not change more than 0.2 percent.

The elemental composition of each insert was determined by the Inductively Coupled Plasma-Atomic Emission Spectra Method performed by PNL. Samples were fused with both K and Na/Zr hydroxides to obtain most of the elemental concentrations. The oxygen, hydrogen, and carbon contents could not be obtained with this analysis. The results of these analyses are shown in Table G.3.

The carbon content can be inferred from the amount of boron present, since it can be assumed that this element normally is not present. From the boron contents shown in Table G.3, the B_4C content in the 0% B_4C, 1% B_4C, 2% B_4C, and 6% B_4C are 0.0, 0.72, 1.97, and 6.39 wt%, respectively. These evaluations accounted for the 0.02 wt% naturally occurring boron in concrete.

The remaining elements in the concrete inserts are oxygen and hydrogen. The hydrogen is assumed to be associated with the water content in the concrete. Approximately 7 g samples of pulverized concrete from each of the inserts were dried in incremental steps to 1000°C. After heating to a given temperature, the sample was cooled in a desiccator and then weighed. The results of this analysis are shown in Table G.4. The PNL staff conducting the measurements observed the following during the analysis: At 750°C, the samples containing B_4C showed a reddish tint with the 6% B_4C sample showing a significant reddish color. At 1000°C, the sample with 6% B_4C had definitely glassified while the sample with 2% B_4C showed some characteristic of forming a glass. From the table of weight changes with temperature, the 2% B_4C and 6% B_4C samples showed a definite weight gain at 750°C. This weight change and color change is probably due to the decomposition of B_4C to B_2O_3 and CO_2. The reddish color noted in the samples at this temperature is probably due to the formation of Fe_2O_3. Also, if CO_2 is formed in cement, this gas is probably trapped as $CaCO_3$ by the CaO in the cement. With a source of B_2O_3 from the B_4C decomposition, a borosilicate-type glass forms at about 1000°C.

The amount of water in the concrete can be deduced from the data in Table G.4. Since the samples are both losing weight by water evaporation and gaining weight by material decomposition, some assumptions are necessary to evaluate the total water content in each of the inserts.

The amount of oxygen in the inserts can be deduced by subtracting the elemental compositions in Table G.3, and the carbon and hydrogen contents from 100.

TABLE G.1 Mixing Components Used to Fabricate Concrete Inserts

Component	Insert			
	0% B_4C (lb)	1% B_4C (lb)	2% B_4C (lb)	6% B_4C (lb)
Water	28.16	27.42	27.86	27.57
Cement	56.22	56.22	55.47	56.22
Aggregate	154.60	155.00	150.00	150.00
B_4C	0.00	1.55	4.64	15.45

TABLE G.2 Certificate of Analysis for B_4C

NOAH CHEMICAL DIV. NOAH INDUSTRIAL CORP.

87 GAZZA BOULEVARD, FARMINGDALE, NEW YORK 11735 TELEPHONE (516) 293-3336 TELEX 645175

Certificate of Analysis

P.O. no. 78720 AD

PRODUCT: BORON CARBIDE, B_4C, 99.5%, -325 mesh

LOT: 37855

ANALYSIS: Average particle size by Fisher = 5.95 microns

C	(theo.)	=	21.74%
C	(found)	=	22.57%
B	(theo.)	=	78.26%
B	(found)	=	78.0%

Al	0.01%
Ca	0.01
Cu	0.005
Fe	0.06
Mg	5 ppm
Mn	5 ppm
Si	.04%
Sn	5 ppm
Zn	.004%

TABLE G.3 Concentration of Elements in Annular Cylinder Concrete Inserts

Element	Insert Composition (wt%)			
	0% B_4C	1% B_4C	2% B_4C	6% B_4C
Al	4.82	4.90	4.82	4.53
B	0.02	0.58	1.56	5.02
Ba	0.07	0.07	0.07	0.06
Ca	15.4	15.3	13.9	14.0
Cu	0.01	0.01	0.01	0.01
Fe	4.39	4.47	4.35	4.16
K	0.54	0.64	1.52	1.36
Li	0.02	0.01	0.02	0.02
Mg	1.12	1.15	1.15	1.14
Mn	0.07	0.07	0.07	0.07
Na	1.89	1.59	1.48	1.45
Si	21.8	22.4	22.1	20.6
Sr	0.04	0.04	0.03	0.03
Ti	0.57	0.58	0.58	0.55
Zr	0.04	0.06	0.10	0.04

TABLE G.4 Analysis for Water Content of Concrete Inserts

Temperature (°C)	Concrete Sample Weight (g)			
	0% B_4C	1% B_4C	2% B_4C	6% B_4C
25	7.8207	8.8389	6.8866	7.4325
110	7.5881	8.6029	6.7275	7.3098
250	7.4781	8.4858	6.5091	7.2027
500	7.3459	8.4039	6.4549	7.2311
750	7.1731	8.2570	6.4736	7.6231
1000	7.1554	8.2312	6.4499	7.5940

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