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**Critical Experiments With
Aqueous Solutions of
 $^{233}\text{UO}_2(\text{NO}_3)_2$**

J. T. Thomas
C. M. Hopper

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Computational Physics and Engineering Division (10)

**Critical Experiments With Aqueous Solutions
of $^{233}\text{UO}_2(\text{NO}_3)_2$**

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ABSTRACT

This report provides the critical experimenter's interpretations and descriptions of informal critical experiment logbook notes and associated information (e.g., experimental equipment designs/sketches, chemical and isotopic analyses, etc.) for the purpose of formally documenting the results of critical experiments performed in the late 1960s at the Oak Ridge Critical Experiments Facility. The experiments were conducted with aqueous solutions of 97.6 wt % ^{233}U uranyl nitrate having uranium densities varying between about 346 g U/l and 45 g U/l. Criticality was achieved with single simple units (e.g., cylinders and spheres) and with spaced subcritical simple cylindrical units arranged in unreflected, water-reflected, and polyethylene reflected critical arrays

ACKNOWLEDGMENT

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1. INTRODUCTION

Criticality experiments utilizing aqueous solutions of uranyl nitrate having a high ^{233}U content were initiated in August of 1966 and continued to early April of 1968. The experiments were performed at the Oak Ridge Critical Experiments Facility (ORCEF) of the Oak Ridge National Laboratory (ORNL). The work was designed to provide information directly applicable to nuclear criticality safety practices and support analyses of basic geometries. Preliminary results appeared in an annual progress report¹ of the Neutron Physics Division of ORNL. This writing provides interpretations of the experimental configurations in the delayed and supercritical states recorded in the logbook.² The information in the logbook is incomplete for interpreting and documenting the experiments. Relevant associated files were used when available.

1.1. URANYL NITRATE SOLUTIONS

Five shipments of the uranyl nitrate solution were received from August 22, through December 20, 1966. The content of ^{232}U decay products (primarily, ^{212}Pb , ^{212}Bi , and ^{208}Tl) was sufficient to produce radiation fields in excess of 1 R/hr at the surface of some transfer bottles. In mid May 1967, the solution was reprocessed by the Chemical Technology Division of ORNL and returned in early June. Table 1 provides the available information for pre- and post-purified solutions indexed by sample number for the associated experiments, uranium concentration, solution density, and the free nitric acid expressed as normal solution.

The solution was utilized to examine basic safety conditions for storage and transfer. Three samples of the solution were taken during March and April 1967, before the solution was reprocessed to reduce the radiation field in the operations area. Results of the analyses are shown in Table 1 under the subheading pre-purified. There is not a complete description of properties for this solution. Some of the information for the post-purification solutions does not correlate with the data: these are indicated by bold entries. Coulometric analysis provided the uranium concentration as $^{\text{nat}}\text{U}$. This value was corrected by a ratio of the molecular mass of ^{233}U in the solution to that of $^{\text{nat}}\text{U}$. The chemical analyses for the solutions, as recorded in the sample logbook,³ are available in the Appendix.

A least-squares-fit, of the “post-purified” uranium concentration and solution density data in Table 1, is presented in Figure 1. The fit excludes inconsistent data shown with bold entries. Based upon limited laboratory reported nitric acid normality, the atomic ratio of nitrogen-to-uranium (N/U) ranges from 2.69 to 2.85 (excluding bold entries) a trend of increasing with dilution that should not have been present. The excess nitrogen content of the pre-purified solution is not known. Because the excess nitrogen content of the solutions was inconsistent and incomplete, an alternative method for inferring the excess nitric acid content was found. That referenced method is provided in the Appendix to this report.

Table 1. Solution specifications

Sample No.	Experiment reference	Uranium density (mgU/ml)	Solution density (g/ml)	Nitric acid normality
Pre-purified^a				
1A	20	332.0	1.4606	
1B	20	333.1	1.4606	
2	35 – 54	342.7	1.4812	
3	–	345.8	1.4724	
Post-purified^b				
11	61 – 100	203.8	1.2781	
12	120 – 124	138.6	1.1935	0.37
13	125 – 148	103.7	1.1846	0.5
14	149	130.7	1.1811	0.4
15	150	116.8	1.1654	0.4
16	152	102.5	1.1422	0.34
17	153	100.8	1.2096	0.36
18a	155 – 168	99.09	1.1372	0.36
18b	155 – 168	99.68	1.1421	
19	170	83.25	1.1166	
20	171	79.65	1.1102	
21	177 – 184	74.58	1.1043	
22	185	51.63	1.0727	
23	194 – 198	44.63	1.0580	
25	205 – 206	200.2	1.2773	
26	208	190.4	1.2507	
27	210	173.7	1.2324	
28	216 – 225	131.6	1.1869	
29	226	133.9	1.1827	
30	227	110.3	1.1524	
31	229 – 235	95.0	1.1381	
32a	241 – 249	47.80	1.0664	
32b	241 – 249	49.69	1.0639	0.24

^aThe inferred N/U atom ratio is 2.19.

^bThe inferred N/U atom ratio is 2.18.

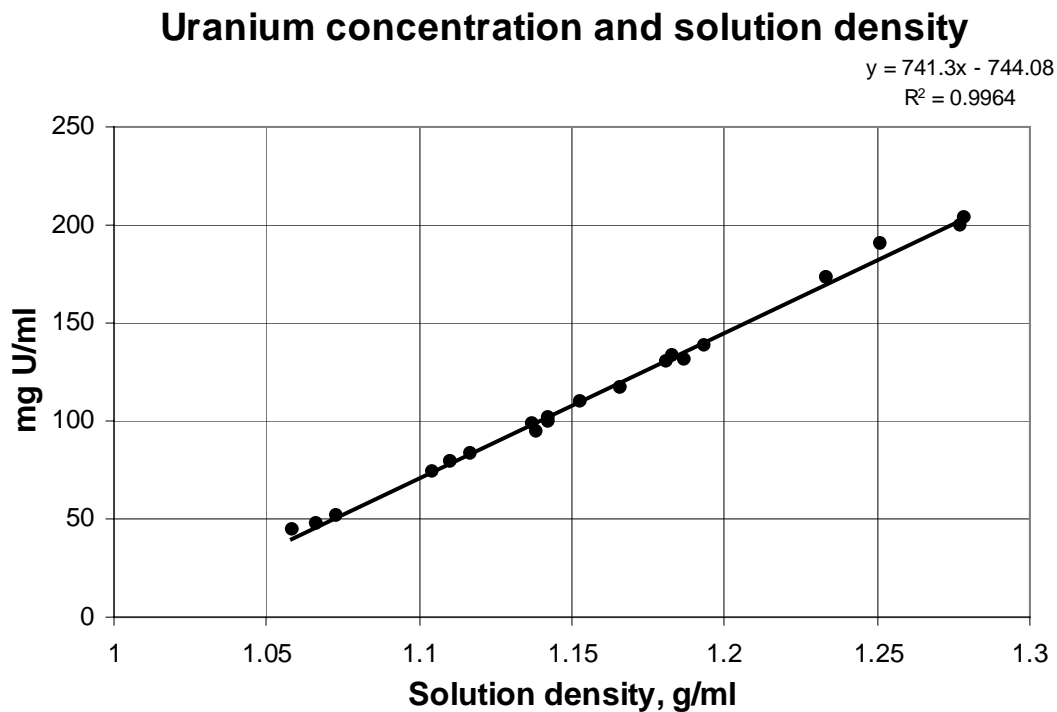


Figure 1. Uranium concentration and solution density.

Table 2 presents isotopic analyses reported on March 18, 1967 for sample No. 2, and a second provided with the returned purified solution on June 9, 1967.

The analyses for impurities present in sample No. 25 are reported in Table 3.

Table 2. Isotopic analyses

Isotope	Pre-purified (wt %)	Purified (wt %)
^{233}U	97.56	97.54
^{234}U	1.053	1.047
^{235}U	0.025	0.026
^{236}U	<0.0005	0.001
^{238}U	1.361	1.386
^{232}U	N/A	6.47 ppm
Average atomic weight	233.1174	233.11863

Table 3. Spectrographic analysis of Sample No. 25

Impurity	Density (mg/ml)	Compound
Al	0.56	Al(NO ₃) ₃
Cr	0.0002	CrO ₃
Cu	0.001	-
Fe	0.003	Fe(NO ₃) ₃
Mn	0.0002	Mn(NO ₃) ₂
Ni	0.001	Ni(NO ₃) ₂
Th	~ 2.5	Th (NO ₃) ₄

The uranium and solution densities are consistent as demonstrated in Figure 1. In that sense, the typical uncertainty for chemical values of $\pm 0.5\%$ would be applicable. However, additional uncertainty is apparent in the data. The duplicates of sample 1 and those of 18 differ by less than 0.6%. Samples 28 and 29 are of the same solution; dilution by demineralized water had not occurred. Twenty-two days elapsed between the sampling dates. The concentrations of uranium differ by 1.5%. Similar behavior is demonstrated in samples 32A and 32B. These samples were taken at the same time; 32A was sent for analysis the day of solution sampling; 32B was sealed under prescribed long-term storage procedures and sent for analysis 19 days later. The uranium concentrations differed by 3.8%.

2. SPACED SUBCRITICAL COMPONENTS

Subcritical units of nearly identical cylindrical volumes of solution were assembled into air-spaced arrays with and without a 15.2-cm-thick polyethylene reflector. The experiments were performed with an equal number of units along the three principle axes of the assemblies, forming 8- and 27-unit arrays. The Criticality Testing Unit, Horizontal Displacement (CTUHD) (a Split Table) apparatus was utilized to provide for partial assembly of an array on the fixed and moveable tables and for controlled remote assembly. The spacing of units was adjusted to define equal separation of the contained solution surfaces in the three axial directions at table closure.

The containers for the fissile solution were fabricated from 0.254-mm-thick stainless steel to the specifications shown in Figure 2. The measured outside diameter and height of eight of the cylinders averaged 18.282 and 17.672-cm, respectively; the 0.254-mm-thick metal defines an 18.231-cm inside diameter; and, the corresponding area is 261.05 cm^2 . Measurements of the height change when the last two liters were added to the cans during their second filling gave an average area of 261 cm^2 . The measured capacity of the containers was 4.630 ± 0.01 liters. The average empty weight of the 27 containers used in the arrays was $324.1 \pm 0.8 \text{ g}$.

The CTUHD was located in the South Assembly Area (Room 113). The west (reference building north) edge of the split table was approximately 3 meters from the west wall of the assembly area, and the north edge approximately 2 meters from the north wall (see Appendix for First and Second Floor Plans of ORCEF). The assembly area was nearly cubic with dimensions of width, height, and depth of approximately 914 cm. All sides were concrete 60.96 cm ceiling, 152 cm west and north wall, 60.96 cm east wall, 91.4 cm south wall, 30.5 cm south corridor wall, and approximately 30.5 cm floor on earth. The top surface of the cast iron bench plates^a of the CTUHD (121.92×182.88 -cm moveable and a 182.88×182.88 -cm stationary) was approximately 2.54 cm thick and 76.2-cm above the floor. On top of each of the tables were four tiers of 7.62-cm-square tubing fabricated with 0.119-cm-thick type 1100 aluminum, 91.44-cm long, and extending the full width of the tables (see Figure 3). A 0.635 cm-thick aluminum 91.44×182.88 -cm sheet was placed on top of each stack of square tubing, making an elevated area 182.88×182.88 -cm when the tables were closed. This served as the base support for the polyethylene-reflector surrounding the arrays and was present in the unreflected array experiments. The same reflector blocks, used in previous experiments,⁴ had a measured density of $0.92 \text{ g polyethylene/cm}^3$. The reactivity contribution of the structure to the assemblies was not measured experimentally. The effect could range from a small fraction to more than half the total reactivity-contribution of the delayed neutrons, depending on the reflector conditions.

The unreflected arrays were constructed using 0.476-cm-diam stainless steel rod for the support grid, with 0.159-cm-thick aluminum plates serving as the base for each fissile unit. The rods were secured to aluminum unistrut attached to a support structure on the tables. The aluminum plates were spaced on the rods by close-fitting Inconel tubing of machined lengths. These experimental features appear in the view of a partially assembled 27-unit array shown in

^a Bench plates are iron castings having one surface machined for flatness. The moveable plate was 121.92×182.88 -cm; the fixed table consisted of two 91.44×182.88 -cm plates.

Figure 3. As scaled from the figure, the spacing from the top surface of the square tubing stack to the bottom of the nearest cylinder was about 38 cm. A sketch of the support plate is given in the Appendix.

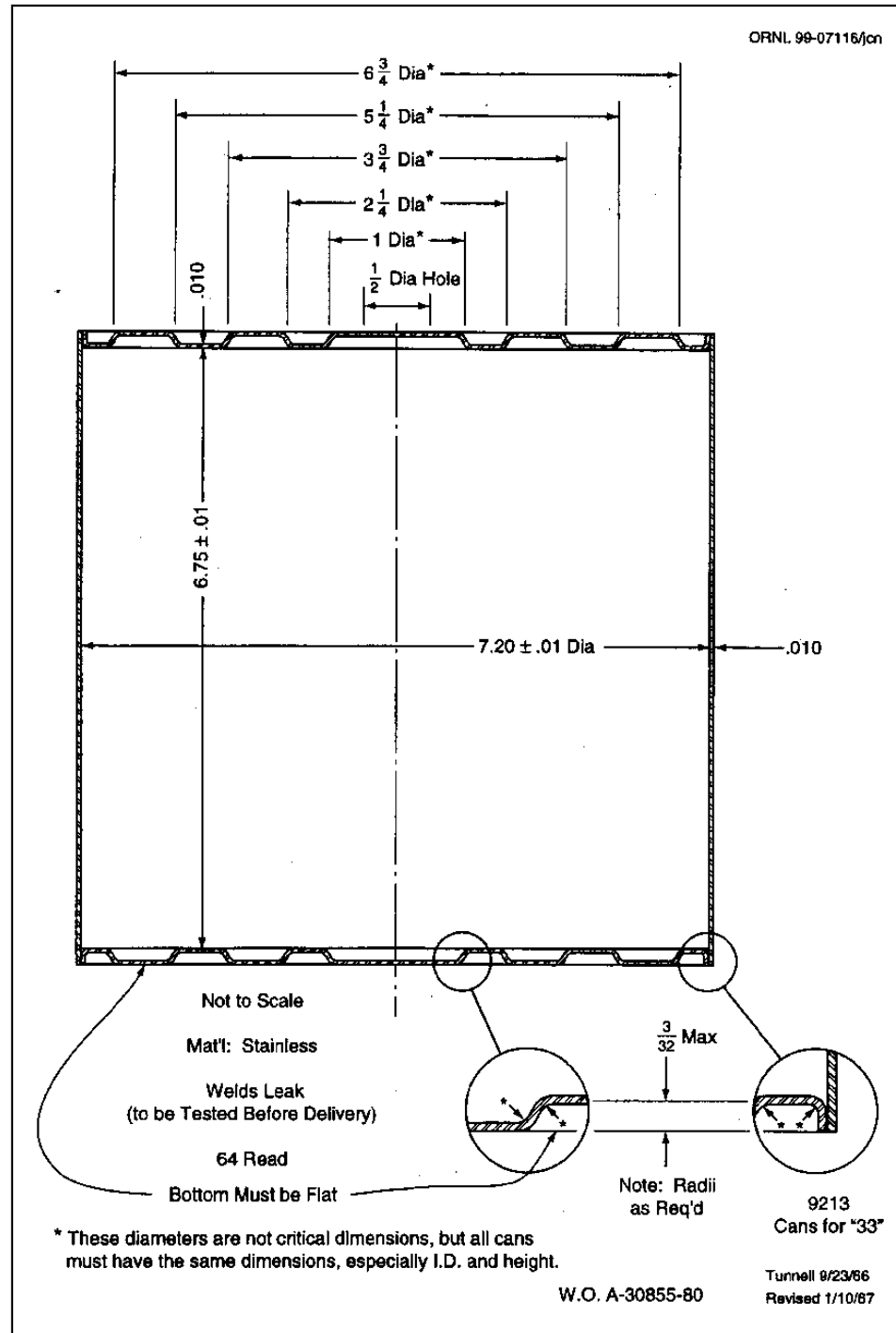


Figure 2. Details of stainless steel solution containers (dimensions in inches).

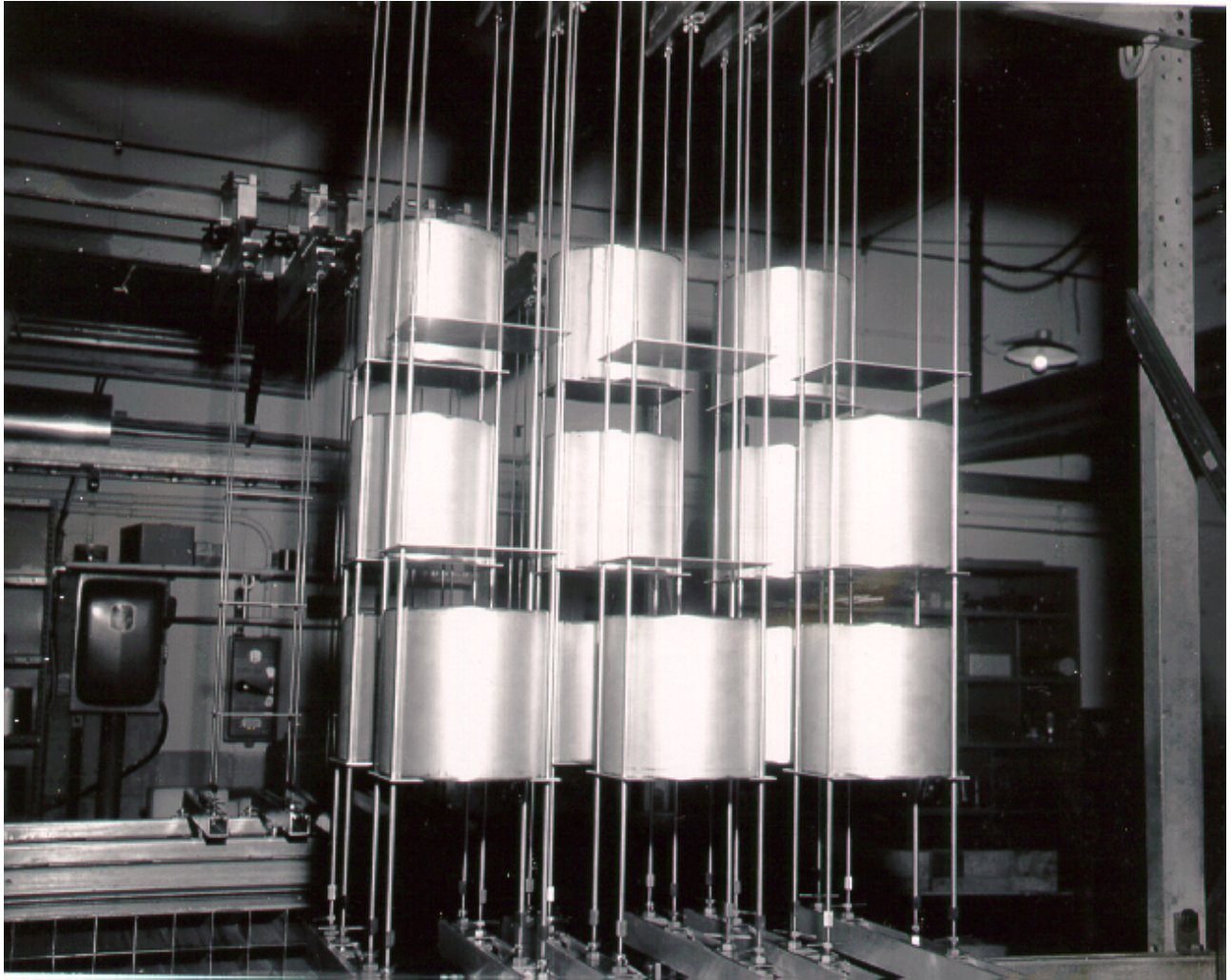


Figure 3. A photograph of a partially assembled 27-unit array taken as viewed in the direction of North.

The fissile units in the reflected arrays were arranged on 0.159-cm-thick aluminum trays supported above the polyethylene-reflector base by machined aluminum channels spaced on threaded rods. The photograph of experiment 85, shown in Figure 4, exemplifies the construction. The specifications for the trays and their support structure appear in a sketch also given in the Appendix. In general, the reflector was located at a boundary defined by the peripheral cell dimensions. An exception to this occurred in experiments 80 through 92 where the trays and supporting Unistrut restrict the xy-plane dimensions of the reflector cavity to a 114.3-cm square.

Experiments performed with fissile solution described by sample No. 2 of Table 1 had an average weight of 6319.9 ± 0.2 -g for the solution. The critical configurations of the containers are presented in Table 4. The headings of the columns present the surface separation of the cylinders in the three coordinate directions when centered in the corresponding cell dimensions. The error in the spacing of the containers was ± 0.02 -cm and ± 0.03 -cm for the unreflected and reflected arrays, respectively. The terms and orientation utilized in the table are depicted in Figure 5, as well as the identity and specific location of the individual fissile cylinders in experiments 44 (a safety confirmation) and 45. With the exception of experiment 37, at table closure, the split between the two tables is located at cell boundaries with units centered horizontally. A measurement of table separation was provided by a linear selsyn (± 0.006 cm) in the control room. The individual fissile material loading of the cylinders and their location in experiments are summarized in the Appendix.

Experiments performed with fissile solution described by sample No. 11 of Table 1 had an average weight of 5551.8 ± 0.8 -g for the solution. The recorded critical configurations of the containers are reproduced in Table 5. Details of solution weight in containers and positions of these containers in arrays also appear in the Appendix.

The critical systems described in Tables 4 and 5 had a k_{eff} of $1.0 \pm 2\%$ of the reactivity interval between delayed and prompt criticality.



Figure 4. Photograph of the fixed table during Experiment 85, a polyethylene reflected 27-cylinder array.

Table 4. Critical array assemblies conducted with solution of Sample No. 2

Expr. No.	E-W	N-S	Table	Vertical	Cell dimensions			Period	Notes	
	delta 1	delta 2	separation	delta 3	y	x	z	T (sec)		
Unreflected 8-unit arrays										
35	1.905	1.905	1.199	0.903	20.187	20.187	18.575	∞	1	
36	2.064	2.064	0.737	1.142	20.346	20.346	18.814	∞		
37	2.381	2.064	0.457	1.142	20.664	20.664	18.814	∞		
38	2.103	2.103	0.038	1.459	20.385	20.385	19.131	∞		
38	2.103	2.103	0.000	1.459	20.385	20.385	19.131	253.1		
Reflected 8-unit arrays										
39	9.287	9.287	11.054	8.207	27.569	27.569	25.879	∞		
40	10.398	10.398	7.760	9.792	28.680	28.680	27.464	∞		
41	13.216	13.216	1.538	11.498	31.498	31.498	29.170	∞		
41	13.216	13.216	1.452	11.498	31.498	31.498	29.170	98.3		
42	13.216	13.216	0.777	12.292	31.498	31.498	29.964	∞		
42	13.216	13.216	0.701	12.292	31.498	31.498	29.964	140.6		
Expr. No.	E-W	N-S	Table	Vertical	Cell dimensions			Period	Notes	
	delta 1	delta 2	separation	delta 3	y	x	z	T (sec)		
Unreflected 27-unit arrays										
45	6.747	6.747	4.420	6.262	25.029	25.029	23.934	∞		
45	6.747	6.747	4.343	6.262	25.029	25.029	23.934	246.6		
46 / 47	7.382	7.382	0.000	6.897	25.664	25.664	24.569	∞		
Reflected 27-unit arrays										
50	21.547	21.547	3.480	20.913	39.829	39.829	38.585	∞		
50	21.547	21.547	3.302	20.913	39.829	39.829	38.585	85.8		
51	22.023	22.023	1.372	21.389	40.305	40.305	39.061	∞		
51	22.023	22.023	1.194	21.389	40.305	40.305	39.061	86.92		
52	22.023	22.023	1.349	21.389	40.305	40.305	39.061	∞		
52	22.023	22.023	1.194	21.389	40.305	40.305	39.061	107.02		
53	22.023	22.023	1.547	21.389	40.305	40.305	39.061	∞		
53	22.023	22.023	1.270	21.389	40.305	40.305	39.061	45.63		
54	22.023	22.023	1.473	21.389	40.305	40.305	39.061	∞		
54	22.023	22.023	1.270	21.389	40.305	40.305	39.061	76.6		
Notes:										
1 At a table separation of 0.140 cm the N-S spacing is 2.381 cm.										
2. Aluminum sheets placed on units of tiers 1 and 2. (Dimensions: 99.06 × 38.1 × 0.159 and 99.06 × 76.2 × 0.159 cm, moveable and fixed tables, respectively.)										
3. Sheets on tier 1 removed, sheets remain on tier 2.										
All dimensions are in centimeters.										

Table 5. Critical array assemblies conducted with solution of Sample No. 11

Expr.	E-W	N-S	Table	Vertical	Cell dimensions			Period	Notes
No.	delta 1	delta 2	separation	delta 3	y	x	z	T (sec)	
Unreflected 8-unit arrays									
61	1.461	1.461	1.435	0.743	19.743	19.743	18.415	∞	
62	1.461	1.461	0.940	1.060	19.743	19.743	18.733	∞	
63	1.801	1.801	0.328	1.060	20.083	20.083	18.733	∞	
65	1.877	1.877	0.089	1.137	20.159	20.159	18.809	∞	
Reflected 8-unit arrays									
69	10.978	11.295	0.831	10.740	29.577	29.260	28.412	∞	
70	11.295	11.295	0.663	11.058	29.577	29.577	28.730	∞	
71	11.295	11.295	0.732	10.740	29.577	29.577	28.412	∞	
Expr.	E-W	N-S	Table	Vertical	Cell dimensions			Period	Notes
No.	delta 1	delta 2	separation	delta 3	y	x	z	T (sec)	
Unreflected 27-unit arrays									
77	6.731	6.731	0.455	6.061	25.013	25.013	23.733	∞	
77	6.731	6.731	0.376	6.061	25.013	25.013	23.733	122.8	
78	6.731	6.731	0.081	6.220	25.013	25.013	23.892	∞	
78	6.731	6.731	0.000	6.220	25.013	25.013	23.892	> 0	1
79	6.731	6.731	0.064	6.220	25.013	25.013	23.892	∞	
79	6.731	6.731	0.000	6.220	25.013	25.013	23.892	604.1	
Reflected 27-unit arrays									
80	19.480	19.480	1.163	18.792	37.762	37.762	36.464	∞	
80	19.480	19.480	0.907	18.792	37.762	37.762	36.464	74.1	
85	19.589	19.589	1.113	18.904	37.871	37.871	36.576	∞	
85	19.589	19.589	0.338	18.904	37.871	37.871	36.576	14.3	
86	19.589	19.589	1.328	18.904	37.871	37.871	36.576	∞	2
86	19.589	19.589	1.021	18.904	37.871	37.871	36.576	63.0	2
87	19.589	19.589	1.262	18.904	37.871	37.871	36.576	∞	3
87	19.589	19.589	1.021	18.904	37.871	37.871	36.576	85.6	3
88	19.589	19.589	1.229	18.904	37.871	37.871	36.576	∞	4
88	19.589	19.589	1.021	18.904	37.871	37.871	36.576	107.5	4
89	19.589	19.589	1.146	18.904	37.871	37.871	36.576	∞	5
89	19.589	19.589	1.021	18.904	37.871	37.871	36.576	337.9	5
92	19.589	19.589	1.085	18.904	37.871	37.871	36.576	∞	6
92	19.589	19.589	0.833	18.904	37.871	37.871	36.576	82.6	6
Notes:									
<div>1. Period not measured</div> <div>2. Aluminum sheets placed on units of tiers 1 and 2. (Dimensions: $99.06 \times 38.1 \times 0.159$ and $99.06 \times 76.2 \times 0.159$ cm, moveable and fixed tables, respectively.)</div> <div>3. Sheets on tier 1 removed, sheets remain on tier 2.</div> <div>4. Aluminum sheet on tier 2 of moveable table removed; remaining sheet is on fixed table, tier 2.</div> <div>5. Remaining sheet on tier 2 moved to top surface of bottom reflector.</div> <div>6. Repeat of experiment 85.</div>									
All dimensions are in centimeters									

3. SIMPLE GEOMETRIES

The series of experiments was conducted with four sizes of cylinders and six of spheres. Cylinder geometries were used at the unique solution concentration defining criticality for a filled sphere in its environment. Additional experiments were performed to evaluate the reactivity contribution from the reflector condition at the base of some cylinders. These were intended to approximate systems suitable for one-dimensional analyses. The procedure entailed the evaluation of the bottom of the aluminum cylinder in the unreflected geometry and the contribution of the aluminum plate and water below it in the reflected geometry. Displacing the water with Styrofoam (polystyrene, density of 0.028 g/ml) effected an approximate worth of the water. Except as otherwise noted, the unreflected simple-geometry solution vessels were not installed within a reflector vessel. Temperature variations in some systems were also explored.

The experiments were conducted in the south assembly area of the ORCEF on the platform shown in Figure 6, a photograph from an earlier program. The platform was relocated against the east wall for the ^{233}U experiments. The solution storage and transfer systems were below the platform and the systems were operated with a closed venting system to reduce the spread of contamination. This is indicated in Figure 7, a schematic of the arrangement. Selsyn readout of the solution height was available in the control room. Minute solution additions could be made to a vessel through the transfer system by the "tad-adder." The "tad-adder" was a 5.08-cm-OD tube having an inside radius of 2.375-cm. and a wall thickness of 0.165 cm that was connected to the transfer system by a flexible U-tube. A selsyn read out of the "tad-adder" also was available in the control room to monitor the vertical position change of the "tad-adder." The readouts were accurate to ± 0.013 cm.

3.1. VESSEL GEOMETRY

3.1.1. Cylinder

The cylinders were fabricated from 2S-aluminum and had a 0.127-cm.-thick wall, a 1.27-cm.-thick flat bottom, and an overall height of 182.88 cm. The cylinders were from the facility's inventory and were constructed in 1952 for use in a series of solution-annuli experiments. The solution fill and drain port is located at the circumference of the cylinder. A sketch of the vessels, Figure 8, presents the dimensions and geometric configuration.

The cylinders were placed in a 45.72-cm radius aluminum vessel for the water-reflected experiments. A sketch of the reflector tank is shown in Figure 9. Note that the reflector tank is not coaxial with the solution cylinders.

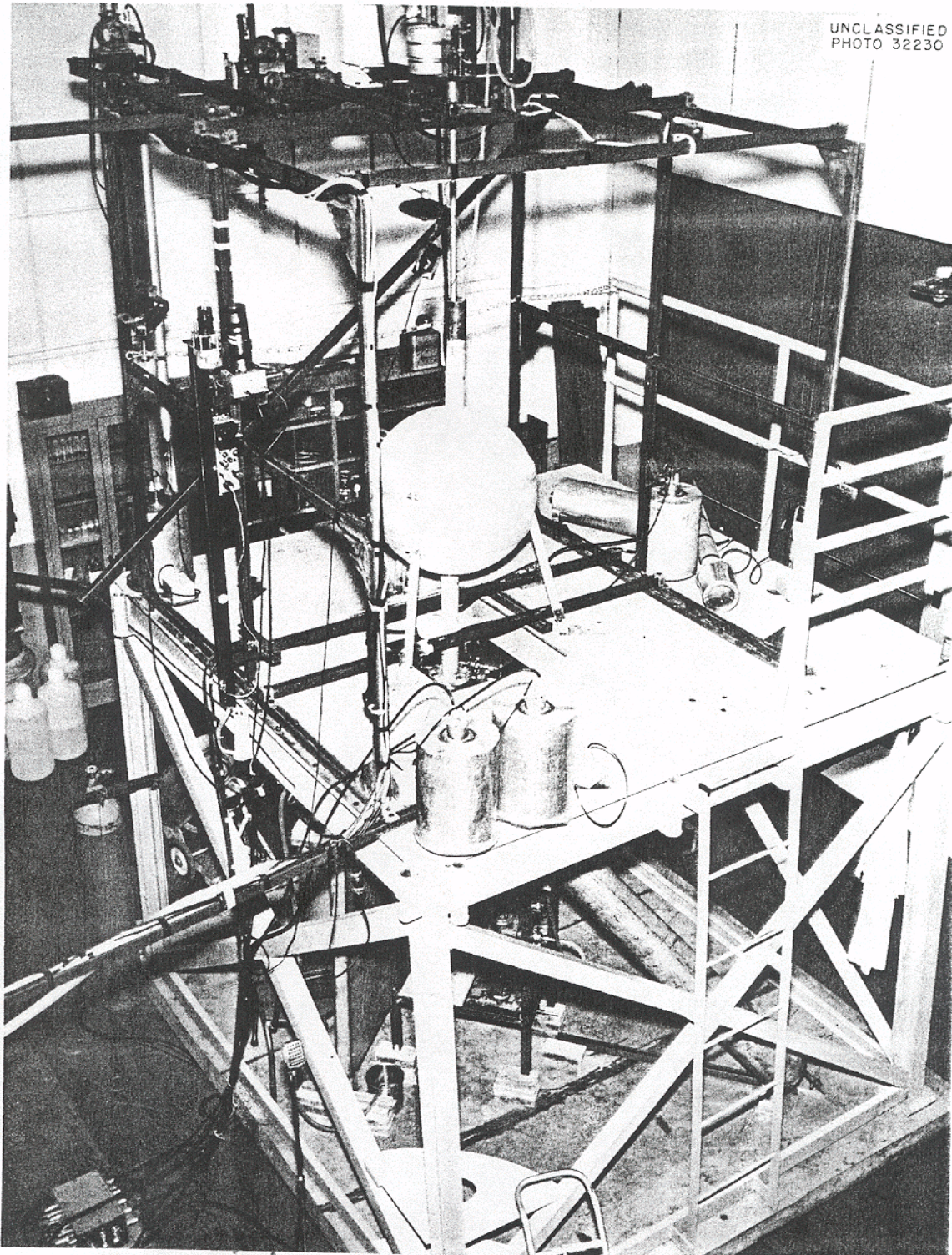


Figure 6. A view of the platform used for the simple geometry experiments. The truck doors shown in the upper-right quadrant are in the east wall of the south assembly area in the ORCEF.

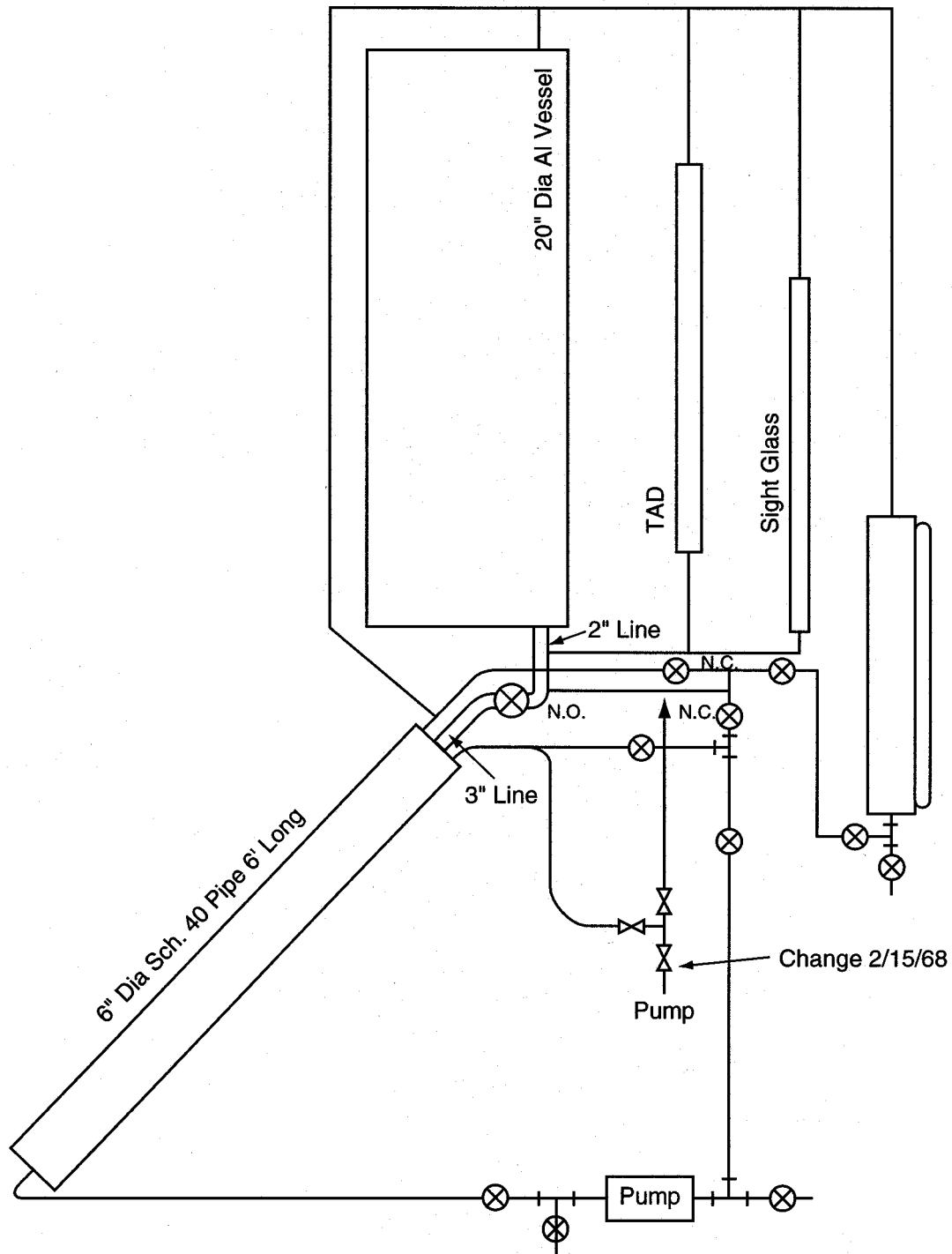


Figure 7. A schematic of the platform showing the relative positions of the storage system, experimental assembly, and the connecting solution lines.

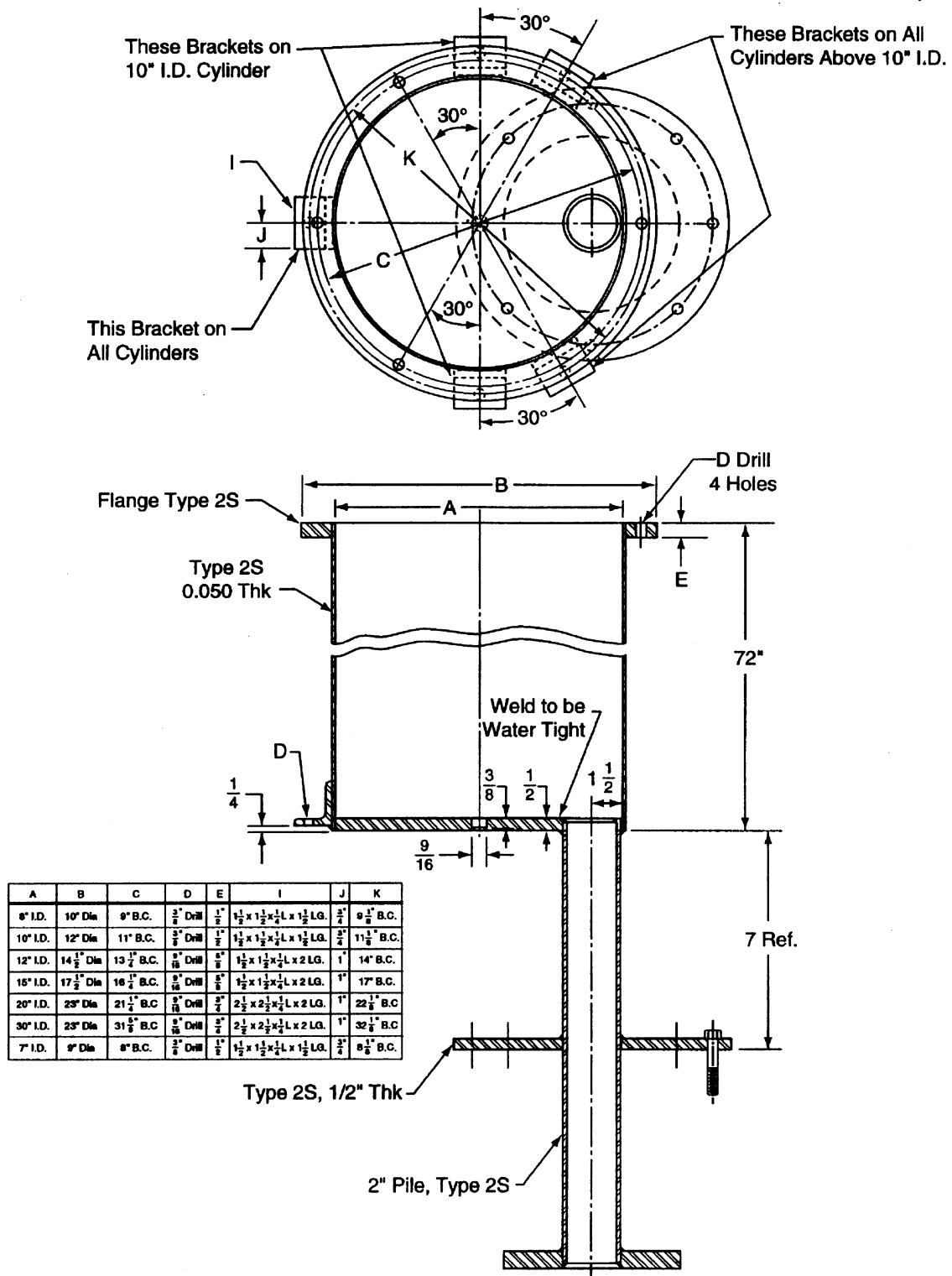


Figure 8. Cylindrical vessels.

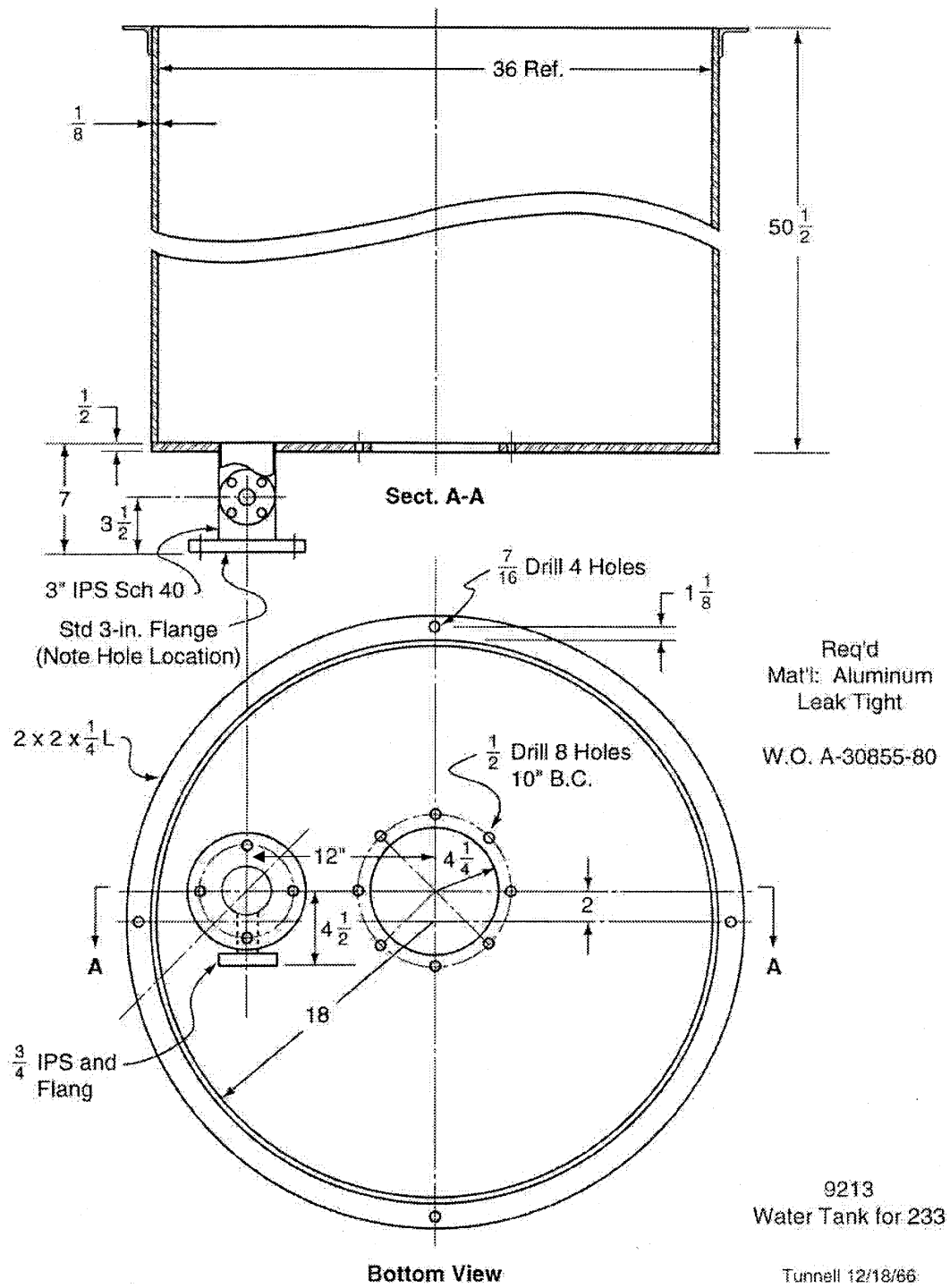


Figure 9. Reflector tank.

neutron level to override the background radiation field. The systems were made supercritical on a stable period until a power level between one and two orders of magnitude above the background was attained. Delayed critical was established and confirmed by a constant power level. In many cases the positive, and sometimes negative, period was measured; relatively few were measured for the water-reflected systems.

Table 8 presents the information describing criticality of the unreflected vessels. The first column records the experiment number. The next three reproduce the logbook entries. The three columns under the heading ‘solution’ give the height corrected for the selsyn zero reading, the volume of solution added to produce a period, and the solution temperature. Experiments having no solution temperature data were at ambient temperature, typically between 21°C and 26°C depending upon the season of the year. The following column is the period associated with the “delta” solution column. The last column provides remarks relevant to the system environment or purpose of the measurement.

The recorded data for the water-reflected vessels are presented in Table 9, which has the same format as Table 8 with an additional column to display the water height. Note that the water height is referenced from the inside bottom of the cylinders.

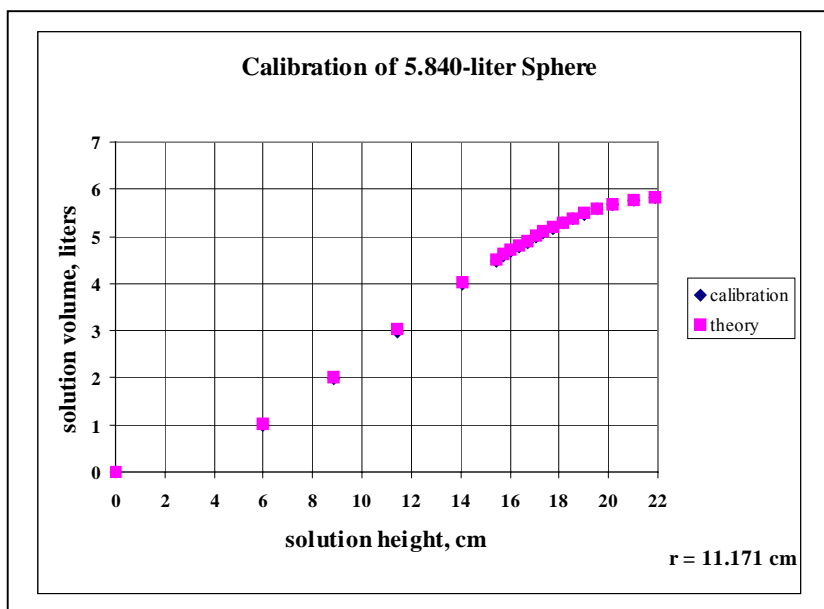
Table 7. Summary of spherical vessel calibration data

Sphere capacity (liters)	Sphere radius (cm)
5.840	11.171
6.964	11.874
12.953	14.569
14.360	15.078
16.450	15.778
26.000	18.378

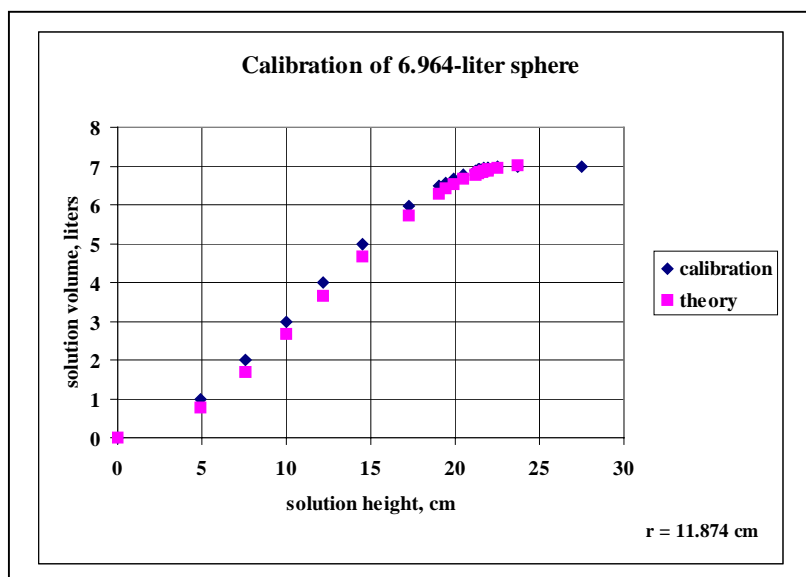
SPHERE CALIBRATIONS

Height (cm)	Volume	
	(liters)	Theory
0	0	0
5.95	0.993	1.022
8.85	1.990	2.023
11.45	2.987	3.029
14.06	3.984	4.026
15.45	4.482	4.514
15.75	4.582	4.614
16.05	4.681	4.710
16.39	4.781	4.816
16.7	4.881	4.909
17.05	4.980	5.011
17.37	5.080	5.100
17.75	5.179	5.200
18.14	5.279	5.296
18.55	5.378	5.391
19	5.478	5.485
19.55	5.577	5.587
20.15	5.677	5.680
21	5.776	5.777
21.9	5.840	5.831

Height (cm)	Volume	
	(liters)	Theory
0	0	0
4.9	0.994	0.772
7.6	1.991	1.695
9.98	2.988	2.674
12.2	3.986	3.651
14.5	4.983	4.651
17.25	5.980	5.725
19	6.478	6.284
19.45	6.577	6.407
19.94	6.677	6.530
20.48	6.776	6.651
21.18	6.875	6.784
21.4	6.900	6.821
21.45	6.910	6.828
21.6	6.925	6.851
21.7	6.936	6.865
21.9	6.949	6.892
21.97	6.954	6.901
22.54	6.966	6.960
23.72	6.972	7.013
27.5	6.993	

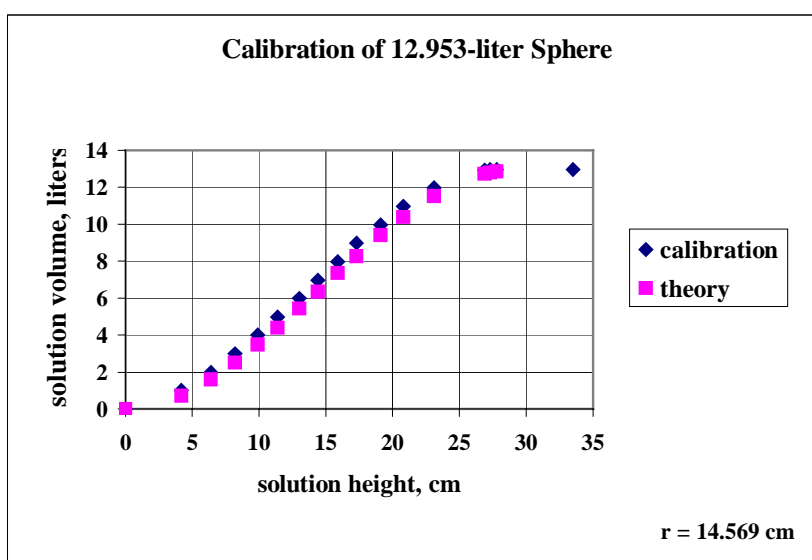


The incremental volume additions and corresponding heights produce the "calibration" label. Volume radius of sphere and the measured heights of accumulated increments produce the "theory" label.



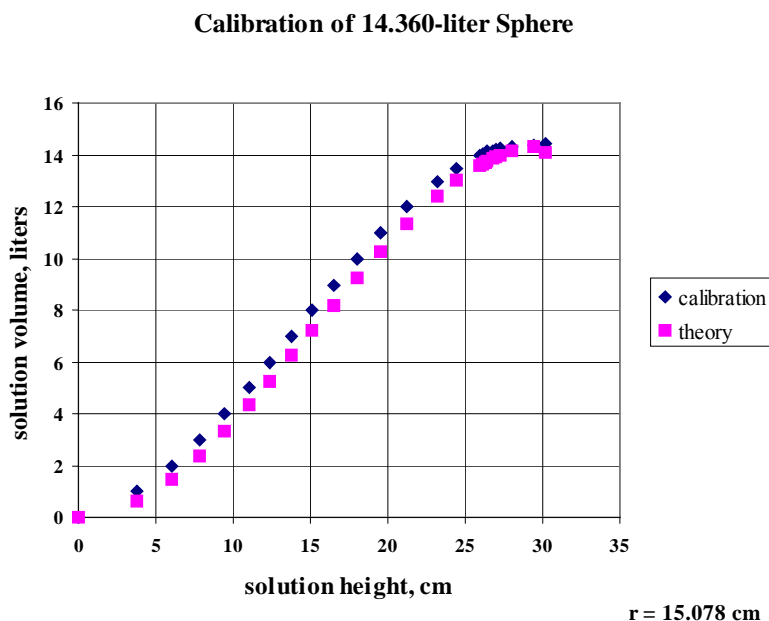
SPHERE CALIBRATIONS

Height (cm)	Volume	
	(liters)	Theory
0	0	0
4.2	0.995	0.730
6.4	1.993	1.600
8.2	2.990	2.500
9.9	3.989	3.470
11.4	4.987	4.397
13	5.985	5.434
14.4	6.983	6.364
15.9	7.982	7.362
17.3	8.980	8.276
19.1	9.978	9.400
20.8	10.976	10.378
23.1	11.973	11.515
26.9	12.919	12.736
27.3	12.943	12.805
27.8	12.953	12.874
33.5	12.961	



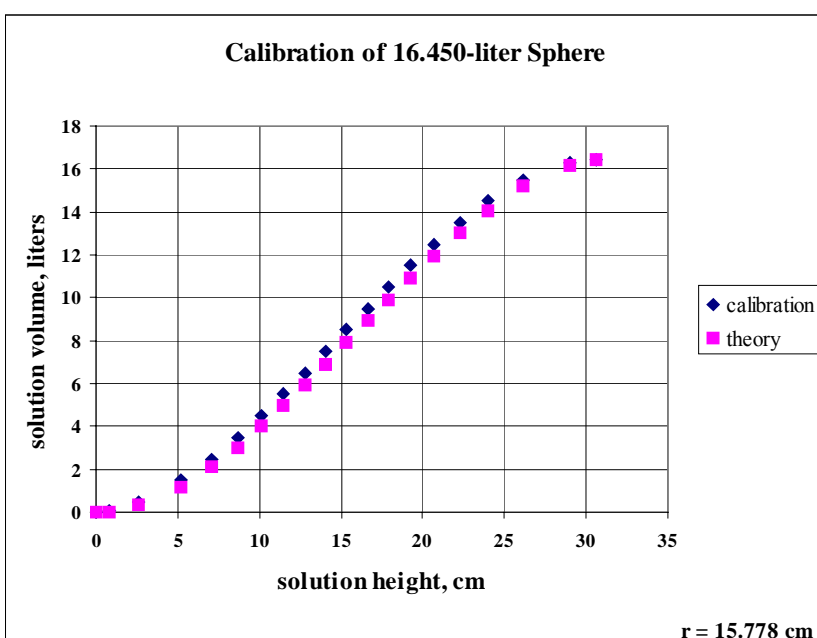
The incremental volume additions and corresponding heights produce the "calibration" label. Volume radius of sphere and the measured heights of accumulated increments produce the "theory" label.

Height (cm)	Volume	
	(liters)	Theory
0	0	0
3.8	0.995	0.627
6.0	1.993	1.479
7.8	2.991	2.385
9.5	3.989	3.347
11.0	4.987	4.338
12.4	5.985	5.252
13.8	6.984	6.234
15.1	7.982	7.195
16.5	8.980	8.192
18.0	9.978	9.241
19.5	10.977	10.248
21.2	11.975	11.312
23.2	12.972	12.420
24.4	13.471	12.990
25.9	13.969	13.582
26.1	14.019	13.650
26.3	14.068	13.715
26.5	14.118	13.762
26.8	14.168	13.852
27.0	14.218	13.910
27.3	14.267	13.993
28.0	14.316	14.150
29.5	14.365	14.337
30.16	14.411	14.098



SPHERE CALIBRATIONS

Height (cm)	Volume	
	(liters)	Theory
0	0	0
0.83	0.086	0.034
2.58	0.485	0.312
5.17	1.486	1.180
7.03	2.488	2.086
8.65	3.489	3.031
10.12	4.491	3.991
11.5	5.492	4.963
12.77	6.493	5.902
14.05	7.495	6.880
15.35	8.497	7.892
16.65	9.497	8.908
17.9	10.499	9.876
19.27	11.500	10.913
20.68	12.502	11.937
22.25	13.505	13.004
23.98	14.507	14.063
26.18	15.508	15.183
29	16.315	16.147
30.65	16.443	16.413
36.35	16.500	15.198



The incremental volume additions and corresponding heights produce the "calibration" label. Volume radius of sphere and the measured heights of accumulated increments produce the "theory" label.

Height (cm)	Volume	
	(liters)	Theory
0	0	0
5.9	2.000	1.795
8.7	4.000	3.680
11	6.000	5.592
13.1	8.000	7.554
15	10.000	9.456
16.9	12.000	11.435
18.8	14.000	13.448
20.7	16.000	15.451
22.6	18.000	17.401
24.7	20.000	19.444
27	22.000	21.478
29.8	24.000	23.559
31.7	25.000	24.660
31.9	25.100	24.759
32.1	25.200	24.855
32.4	25.300	24.992
32.6	25.400	25.079
32.9	25.500	25.202
33.2	25.600	25.318
33.6	25.700	25.458
33.9	25.800	25.554
34.4	25.900	25.694
34.9	26.000	25.808
39.0	26.100	25.698

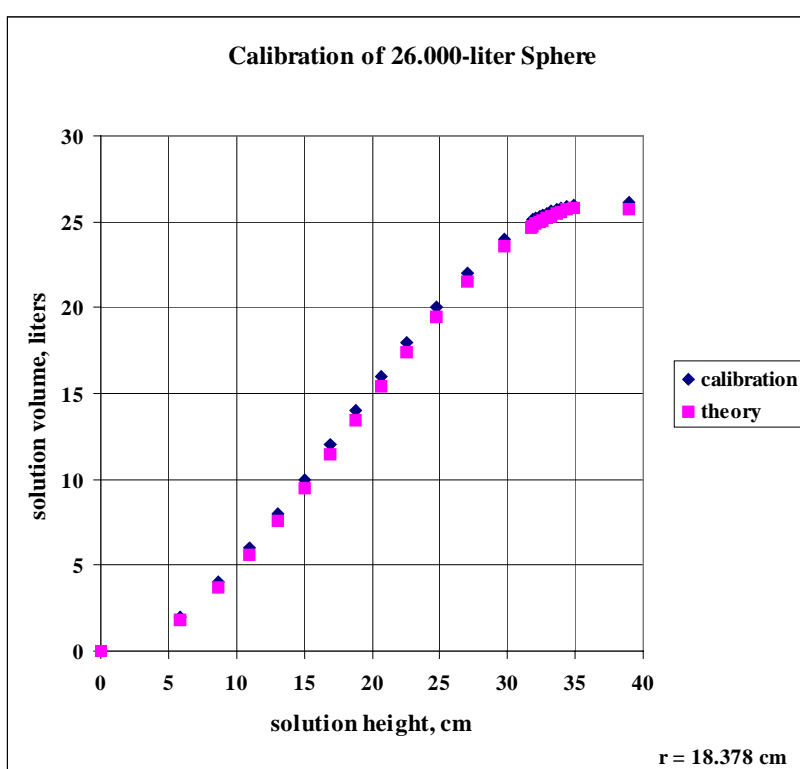


Table 8. Unreflected simple geometries

Expr. No.	Selsyn	Tad (cm)	Sight glass (cm)	Solution		Temp. (°C)	Period T (sec)	Remarks
	zero (cm)			height (cm)	delta* (cm ³)			
Sample No. 1								
Unreflected 20-in.-diam cylinder								2.027 liters/cm
20	0.051	80.086	13.373	13.322	331.501		155.8	
	0.051	61.214	13.360	13.310	0		∞	
Sample No. 11								
93	0.127	79.908	13.614	13.487	379.687		51.7	
	0.127	58.293	13.614	13.487	0		∞	
94	0.127	21.600	13.589	13.462	12.225		117.3	20" × 20" × 1/16" Al sheet
	0.127	20.904	13.589	13.462	0		∞	
95	0.127	24.409	13.538	13.411	15.839		93.98	18" × 20" × 1/8" Al sheet
	0.127	23.508	13.538	13.411	0		∞	
96	0.127	30.612	13.589	13.462	41.137		79.1	repeat experiment 95
	0.127	28.270	13.564	13.437	0		∞	
97	0.127	16.739	13.614	13.487	11.377		149.9	repeat experiment 93
	0.127	16.091	13.614	13.487	0		∞	
Unreflected 15-in.-diam cylinder								1.141 liters/cm
98	0.038	19.507	15.189	15.151	12.853		76.5	
	0.038	18.771	15.189	15.151	0		∞	
100	0.038	17.272	15.189	15.151	13.296		83.2	15" × 15" 1/16" Al sheet
	0.038	16.510	15.164	15.126	0		∞	
101	0.038	16.535	15.164	15.126	14.182		78.9	15" × 15" 1/8" Al sheet
	0.038	15.723	15.113	15.075	0		∞	
102	0.038	16.129	15.202	15.164	20.608		44.8	15" × 15" 1/16" Al sheet
	0.038	14.948	15.154	15.116	0		∞	
Unreflected 10-in.-diam cylinder								0.508 liters/cm
103	0.165	16.993	24.917	24.752	11.308		160.35	
	0.165	16.332	24.867	24.702	0		∞	
104	0.165	16.942	24.905	24.740	21.312		70.16	10" × 10" × 1/16" Al sheet
	0.165	15.697	24.816	24.651	0		∞	
105	0.165	17.526	24.867	24.702	27.401		52.13	10" × 10" × 1/8" Al sheet
	0.165	15.926	24.790	24.625	0		∞	
Unreflected 12.953-liter sphere								
109	0.051	11.049	23.292	23.241	5.663		216.74	sphere in reflector tank.
	0.051	10.770	23.266	23.216	0		∞	
110	0.051	22.098	23.388	23.338	9.009		137.15	removed reflector tank
	0.051	21.654	23.317	23.266	0		∞	void present
Solution sample No. 12								
120	0.051	8.115	26.543	26.492	18.678	22.65	56.72	
	0.051	7.061	26.411	26.360	0	22.65	∞	void present
121	0.051	7.557	26.543	26.492	9.001	24.2	158.63	
	0.051	7.049	26.467	26.416	0	24.25	∞	void present
122	0.051	7.366	26.645	26.594	12.152	26.4	103.76	
	0.051	6.680	26.543	26.492	0	26.4	∞	
123	0.051	6.843	26.746	26.695	17.012	29.5	74.75	
	0.051	5.883	26.657	26.607	0	29.5	∞	void present
124	0.051	6.886	26.937	26.886	20.298	32.25	60.63	
	0.051	5.740	26.810	26.759	0	32.25	∞	void present

*Delta refers to the volume of solution added to, or removed from critical systems. In spherical geometry the increment may be large enough to fill a spherical void and enter the vent pipe.

Table 8 (continued)

Table 8 (continued)								
Expr. No.	Selsyn	Tad (cm)	Sight	Solution			T (sec)	Remarks
	zero (cm)		glass (cm)	height (cm)	delta* (cm ³)	Temp. (°C)		
Solution sample No. 13								
125	0.051	13.360	27.496	27.445	10.802	24.2	138.53	
	0.051	12.751	27.394	27.343	0	24.2	∞	
126	0.051	37.414	27.584	27.534	144.921	25.3	125.49	
	0.051	29.235	27.432	27.381	0	25.3	∞	
127	0.051	1.753	27.889	27.838	23.403	27.5	67.8	
	0.051	0.432	27.648	27.597	0	27.5	∞	
128	0.051	9.982	29.439	29.388	32.405	29.3	55.19	
	0.051	8.153	27.991	27.940	20.253	29.3	74.75	
	0.051	7.010	27.788	27.737	0	29.3	∞	
129	0.051	20.333	37.300	37.249	201.134	30.5	157.91	
	0.051	8.981	27.953	27.902	3.871	30.5	478.06	
	0.051	8.763	27.927	27.877	0	30.4	∞	
130	0.051	12.095	27.308	27.257	12.692	22.6	114.63	
	0.051	11.379	27.165	27.115	0	22.6	∞	
131	0.051	6.655	27.635	27.584	19.803	26.6	72.8	
	0.051	5.537	27.445	27.394	0	26.5	∞	
132	0.051	20.358	27.140	27.089	324.271	23.3	67.36	
	0.051	2.057	26.954	26.904	0	23.2	∞	
133	0.051	10.185	28.029	27.978	22.053	29.8	84.75	
	0.051	8.941	27.559	27.508	0	29.8	∞	
Unreflected 15-in.-diam cylinder								
134	0.051	12.502	16.454	16.403	47.067		56.28	15" cyl. unreflected
	0.051	9.804	16.408	16.358	0		∞	
135	0.051	9.268	16.434	16.383	11.035		167.32	15" × 15" × 1/16" Al sheet
	0.051	8.636	16.383	16.332	0		∞	
136	0.051	9.017	16.403	16.353	11.080		134.73	15" × 15" × 1/8" Al sheet
	0.051	8.382	16.373	16.322	0		∞	
Unreflected 20-in.-diam cylinder								
139	-0.025	9.246	14.326	14.351	12.046	23.4	159.35	20" cyl. unreflected
	-0.025	8.560	14.326	14.351	0	23.4	∞	
140	-0.025	12.926	13.960	13.985	11.779	24.1	165.50	20" × 20" × 1/8" Al sheet
	-0.025	12.256	13.960	13.985	0	24.1	∞	
141	-0.025	12.827	14.008	14.034	20.301	24.3	85.59	20" × 20" × 1/16" Al sheet
	-0.025	11.671	14.008	14.034	0	24.4	∞	
142	-0.025	10.488	14.049	14.074	21.371	24.7	80.16	repeat experiment 139
	-0.025	9.271	14.049	14.074	0	24.7	∞	
143	-0.025	11.938	14.049	14.074	19.854	24.5	91.46	repeat experiment 142
	-0.025	10.808	14.049	14.074	0	24.5	∞	
Unreflected 10-in.-diam cylinder								
144	0.025	13.233	28.628	28.603	17.398	24.7	131.08	10 cyl. unreflected
	0.025	12.217	28.562	28.537	0.000	24.7	∞	
145	0.025	12.626	28.651	28.626	37.013	24.8	52.13	10" × 10" × 1/16" Al sheet
	0.025	10.465	28.527	28.501	0.000	24.8	∞	
146	0.025	12.700	28.626	28.600	33.055	24.85	66.90	10" × 10" × 1/8" Al sheet
	0.025	10.770	28.512	28.486	0.000	24.84	∞	
147	0.025	13.233	28.727	28.702	21.312	25.5	115.69	repeat experiment 144
	0.025	11.989	28.639	28.613	0	26	∞	

*Delta refers to the volume of solution added to, or removed from critical systems. In spherical geometry the increment may be large enough to fill a spherical void and enter the vent pipe.

Table 8 (continued)

Selsyn		Sight		Solution			T (sec)	Remarks
Expr. No.	zero (cm)	Tad (cm)	glass (cm)	height (cm)	delta* (cm ³)	Temp. (°C)		
Unreflected 16.6-liter sphere								
168	0.000	17.285	26.772	26.772	21.880	21.6	> 0	16.6 liter sphere
	0.000	16.205	26.759	26.759	0	21.6	∞	void present
Solution sample No. 19								
170	0.000	19.050	26.988	26.988	23.167	22.5	> 0	
	0.000	17.907	26.975	26.975	0	22.5	∞	void present
Solution sample No. 20								
171	0.000	21.692	28.219	28.219	15.959	25.8	> 0	
	0.000	20.904	28.209	28.209	0	25.8	∞	void present
Solution sample No. 21								
177	-4.547	22.987	32.842	37.389	127.674	21	> 0	
	-4.547	16.688	30.963	35.509	0	21.1	∞	
Unreflected 15-in.-diam cylinder								
178	-0.051	7.188	19.213	19.263	17.284	21.3	> 0	15" cyl. unreflected
	-0.051	6.198	19.192	19.243	0	21.3	∞	15 × 15 × 1/8 Al sheet
179	-0.051	28.804	19.355	19.406	12.188	21.4	> 0	
	-0.051	28.105	19.317	19.368	0	21.4	∞	
180	-0.051	26.467	19.284	19.334	13.296	21.5	> 0	15 × 15 × 1/16 Al sheet
	-0.051	25.705	19.279	19.329	0	21.6	∞	
Unreflected 10-in.-diam cylinder								
181	0.102	13.462	43.663	43.561	64.632	21.7	> 0	10" cyl. unreflected
	0.102	9.688	43.632	43.531	0	21.7	∞	10 × 10 × 1/8 Al sheet
182	0.102	43.180	43.790	43.688	89.075	21.8	> 0	
	0.102	37.978	43.739	43.637	0	21.8	∞	
183	0.102	50.571	43.688	43.586	233.562	21.9	> 0	10 × 10 × 1/16 Al sheet
	0.102	36.932	43.675	43.574	0	21.9	∞	
Unreflected 26-liter sphere								
184	0.089	7.734	24.194	24.105	30.426	21.9	> 0	26 liter sphere
	0.089	6.233	24.181	24.092	0	21.9	∞	void present
Solution sample No. 22								
185	0.089	9.677	29.108	29.020	49.422	30.5	> 0	
	0.089	7.239	29.083	28.994	0	30.5	∞	void present
Solution sample No. 23								
194	0.089	11.963	35.141	35.052	61.778	21.3	> 0	
	0.089	8.915	35.027	34.938	0	21.3	∞	
	0.089	12.014	34.976	34.887	62.807	21.3	> 0	
	0.089	8.915	35.065	34.976	0	21.3	∞	
Unreflected 15-in.-diam cylinder								
196	-0.076	13.983	26.226	26.302	55.177	21.3	> 0	15" cyl. unreflected
	-0.076	10.820	26.187	26.264	0	21.3	∞	15 × 15 × 1/8 Al sheet
	-0.076	29.515	26.264	26.340	44.762	21.3	> 0	
	-0.076	26.949	26.251	26.327	0	21.3	∞	
	-0.076	30.767	26.297	26.373	24.509	21.3	> 0	15 × 15 × 1/16 Al sheet
	-0.076	29.362	26.289	26.365	0	21.3	∞	

*Delta refers to the volume of solution added to, or removed from critical systems. In spherical geometry the increment may be large enough to fill a spherical void and enter the vent pipe.

Table 9. Reflected simple geometries

Selsyn		Sight glass (cm)	Solution				Reflector height (cm)	Period T (sec)	Remarks
Expr. No.	zero (cm)		Tad (cm)	height (cm)	delta* (cm ³)	Temp. (°C)			
Solution sample No. 25									
5.840-liter sphere									
205	0	64.084	17.983	17.983	6.661			90.72	
	0	63.708	17.971	17.971	0	22.2	67.5	∞	void present
206	0	35.839	17.945	17.945	7.201			88.01	
	0	35.433	17.958	17.958	0	22.3	68.5	∞	repeat experiment 205
Solution sample No. 26									
208	0	34.112	18.606	18.606	4.95		67.5	146.31	
	0	33.833	18.606	18.606	0	25		∞	void present
Solution sample No. 27									
210	0	33.782	19.538	19.538	6.751	29.95	67.5	95.59	
	0	33.401	19.563	19.563	0	28.75		∞	void present
Solution sample No. 28									
216	0	37.897	21.590	21.590	4.276	25.3	68.5	195.6	
	0	37.656	21.580	21.580	0	25.7	68.5	∞	temperature
217	0	37.668	21.920	21.920	10.802	27	68.5	10.7	
	0	37.059	21.641	21.641	0	27	68.5	∞	temperature
	0	36.576	21.575	21.575	-8.551	27	68.5	-14.6	
218	0	36.576	21.628	21.628	5.851	25.8	68.5	∞	
	0	36.246	21.628	21.628	0	26	68.5	∞	
Water-reflected 15-in-diam. cylinder									
219	-0.0762	37.452	13.492	13.569	0	25	31.5	∞	w/styrofoam on bottom.
	-0.0762	27.965	13.386	13.462	0	25	43	∞	Water height at bottom
	-0.0762	33.769	13.452	13.528	0	25	35.3	∞	of cylinder is 1.3 cm.
220	-0.0762	31.801	13.335	13.411	0	24	37.4	∞	Water thickness below
	-0.0762	33.579	13.386	13.462	0	24	35.3	∞	bottom is 24.16 cm.
221	-0.0762	31.623	11.773	11.849	0	25	31.4	∞	Removed styrofoam from
	-0.0762	31.394	11.760	11.836	0	25	32.8	∞	below the cylinder.
	-0.0762	31.077	11.770	11.847	0	25	33.1	∞	
	-0.0762	30.582	11.763	11.839	0	25	33.5	∞	
	-0.0762	30.074	11.760	11.836	0	25	34.2	∞	
	-0.0762	30.328	11.748	11.824	0	25	34.9	∞	
	-0.0762	29.108	11.748	11.824	0	25	35.4	∞	
	-0.0762	28.600	11.725	11.801	0	25	36	∞	
Water-reflected 10-in-diam. cylinder									
222	0.1016	26.568	17.308	17.206	0	25	43.5	∞	w/styrofoam
	0.1016	26.683	17.310	17.209	0	25	43	∞	Water height at bottom
	0.1016	27.026	17.323	17.221	0	25	42	∞	of cylinder is 1.4 cm.
223	0.1016	28.087	15.583	15.481	0	25	40.1	∞	Removed styrofoam from
	0.1016	28.245	15.596	15.494	0	25	39.8	∞	below the cylinder.
	0.1016	28.463	15.606	15.504	0	25	39.1	∞	

*Delta refers to the volume of solution added to, or removed from critical systems. In spherical geometry the increment may be large enough to fill a spherical void and enter the vent pipe.

Table 9 (continued)

Expr. No.	Selsyn zero (cm)	Tad (cm)	Sight glass (cm)	Solution height (cm)	Solution delta* (cm ³)	Temp. (°C)	Reflector height (cm)	Period T (sec)	Remarks
Water-reflected 8-in-diam. cylinder									
224	0.1016	27.457	22.819	22.870	0	25	48.5	∞	w/styrofoam
	0.1016	27.483	22.819	22.870	0	25	48	∞	Water height at bottom
	0.1016	27.584	22.819	22.870	0	25	47.8	∞	of cylinder is 2.0 cm.
	0.1016	27.661	22.822	22.873	0	25	47.5	∞	
	0.1016	27.965	22.835	22.885	0	25	46.1	∞	
225	0.1016	30.251	21.112	21.163	0	25	46	∞	Removed styrofoam
	0.1016	30.759	21.148	21.199	0	25	43.7	∞	from below the cylinder.
Solution sample No. 29									
6.964-liter-sphere									
226	-0.0762	27.737	17.742	17.818	0	23.7	66	∞	void present
Solution sample No. 30									
227	-0.0762	35.484	20.597	20.673	0	23.8	65.8	∞	void present
Solution sample No. 31									
229	-0.0762	40.640	31.610	31.687	71.491	24.4	65.8	> 0	
	-0.0762	36.627	28.664	28.740	0	24.4	65.8	∞	
Water-reflected 10-in-diam. cylinder									
231	-0.0762	11.760	19.622	19.685	0	25	45	> 0	w/styrofoam
	-0.0762	11.252	19.611	19.675	0	25	45	∞	Water height at bottom
	-0.0762	11.379	19.614	19.677	0	25	44.5	∞	of cylinder is 1.4 cm.
	-0.0762	11.532	19.619	19.682	0	25	44	∞	
	-0.0762	11.760	19.622	19.685	0	25	43.5	∞	
232	-0.0762	11.963	17.859	17.922	0	25	42.6	> 0	Removed styrofoam from
	-0.0762	11.341	17.854	17.917	0	25	42.6	∞	below the cylinder.
	-0.0762	11.455	17.856	17.920	0	25	4.14	∞	
Water-reflected 8-in-diam. cylinder									
233	0.1778	12.014	27.211	27.033	0	25	52	> 0	w/styrofoam
	0.1778	11.303	27.221	27.043	0	25	52	∞	Water height at bottom
	0.1778	11.760	27.206	27.028	0	25	49.9	∞	of cylinder is 2.0 cm.
	0.1778	12.014	179.611	179.433	0	25	48.6	∞	
234	0.1778	10.605	25.606	25.428	0	25	50.9	> 0	Removed styrofoam from
	0.1778	9.474	25.578	25.400	0	25	50.9	∞	below the cylinder.
	0.1778	9.550	25.578	25.400	0	25	50.2	∞	
	0.1778	10.592	25.626	25.448	0	25	45.8	∞	
Water-reflected 12.953-liter sphere									
235	0	22.504	17.379	17.379	7.201	24.5	66	> 0	
	0	22.098	17.369	17.369	0	24.5	66	∞	void present
Solution sample No. 32									
241	0	36.538	38.087	38.087	205.454	27.9	65.8	> 0	
		24.943	28.473	28.473	0	27.5	65.8	∞	void present
242	0	21.057	27.597	27.597	15.302	25	65.8	> 0	
	0	20.193	27.483	27.483	0	25.2	65.8	∞	

*Delta refers to the volume of solution added to, or removed from critical systems. In spherical geometry the increment may be large enough to fill a spherical void and enter the vent pipe.

Table 9 (continued)

Table 3 (continued)									
Selsyn		Tad (cm)	Sight glass (cm)	Solution			Reflector height (cm)	Period T (sec)	Remarks
Expr. No.	zero (cm)			height (cm)	delta* (cm ³)	Temp. (°C)			
Water-reflected 10-in-diam. cylinder									
243	0.1016	19.469	31.623	31.521	0	26	60.5	> 0	w/styrofoam
	0.1016	18.136	31.603	31.501	0	26	60.5	∞	Water height at bottom
	0.1016	18.212	31.603	31.501	0	26	60	∞	of cylinder is 1.4 cm.
	0.1016	18.428	31.603	31.501	0	26	58.9	∞	
	0.1016	19.469	31.636	31.534	0	26	55.5	∞	
244	0.1016	18.847	29.985	29.883	0	26	55	> 0	Removed styrofoam from
	0.1016	17.272	29.959	29.858	0	26	55	∞	below the cylinder.
	0.1016	17.348	29.959	29.858	0	26	54.3	∞	
	0.1016	18.847	29.997	29.896	0	26	50	∞	
Water-reflected 15-in-diam. cylinder									
248	0.0254	10.693	19.583	19.558	0	25	48	> 0	w/styrofoam
	0.0254	9.703	19.583	19.558	0	25	48	∞	Water height at bottom
	0.0254	10.109	19.583	19.558	0	25	47	∞	of cylinder is 1.3 cm.
	0.0254	10.693	19.588	19.563	0	25	46	∞	
	0.0254	11.532	19.606	19.581	0	25	44.7	∞	
	0.0254	12.111	19.614	19.588	0	25	43.7	∞	
249	0.0254	23.851	18.108	18.082	0	26	42	> 0	Removed styrofoam from
	0.0254	23.025	18.098	18.072	0	26	42	∞	below the cylinder.
	0.0254	23.851	18.110	18.085	0	26	41	∞	

*Delta refers to the volume of solution added to, or removed from critical systems. In spherical geometry the increment may be large enough to fill a spherical void and enter the vent pipe.

4. REFERENCES

1. J. T. Thomas, *Critical Experiments With Aqueous Solutions of $^{233}\text{UO}_2(\text{NO}_3)_2$* , pp. 53-55, ORNL-4280, Neutron Physics Division Annual Progress Report Period Ending May 31, 1968.
2. Experiments Logbook “ ^{233}U Tinkertoy,” Scanned Book 5r.
3. “Sample Information Log,” Scanned Book 32r.
4. D. W. Magnuson, *Critical Three-Dimensional Arrays of Neutron Interacting Units: Part III. Arrays of U(93.2) Metal Separated by Various Materials*, Y-DR-83, Oak Ridge Y-12 Plant, Union Carbide Corporation, Oak Ridge, Tennessee, May 15, 1972.

APPENDIX

INTRA-LABORATORY CORRESPONDENCE

OAK RIDGE NATIONAL LABORATORY

To: H. F. Stringfield

Date: June 9, 1967

Subject: Transfer of ^{233}U to Neutron Physics

On this date, I transferred 26.085 kg uranium (25.440 kg ^{233}U) to the Neutron Physics Division. This material was purified the week of June 5, 1967 (Run 67597). The solution was transferred, in 11-liter bottles to the Neutron Physics truck. The attached table gives the weights and quantities in each bottle.

The isotopic analyses, received from Ray Eby by telephone is as follows:

<u>Isotope</u>	<u>Wt %</u>
^{233}U	97.54
^{234}U	1.047
^{235}U	.026
^{236}U	.001
^{238}U	1.386
^{232}U	6.47 ppm

The solution was analyzed as a batch in tank R-25 (coded: Run No. 67597, sample UP-1-1,2). The results, reported on sheet No. 16282 gave the uranium concentration of 208.6 g/liter as ^{238}U . This value was corrected by the mass ratio $234.05/238.04$ which gave a uranium concentration of 204.2 g/liter. By multiplying this value by the isotopic analyses (97.54%), the uranium concentration of 199.2 g/liter was determined.

ORIGINAL SIGNED BY

J. R. PARROTT

J. R. Parrott
Pilot Plant Section
Chemical Technology Division

JRP:bjh

Attachments (2)

cc: R. E. Brooksbank

J. T. Thomas (9213)

W. A. Shannon

H. B. Graham

J. H. Walker

JRP-File

Date: June 9, 1967

Transfer of ^{233}U to Neutron Physics

Bottle No.	Gross Wt. (g)	Tare Wt. (g)	Net Wt. (g)	Volume (ℓ)	Total U (g)	^{233}U (g)	Radiation Reading (mr/hr)
1	16,050	4,184	11,866	9.54	1,948	1,900	80
2	16,250	4,159	12,091	9.72	1,985	1,936	60
3	16,270	4,200	12,070	9.70	1,981	1,932	60
4	16,500	4,369	12,131	9.75	1,991	1,942	60
5	16,550	4,419	12,131	9.75	1,991	1,942	60
6	16,090	4,060	12,030	9.67	1,975	1,926	55
7	16,250	4,159	12,091	9.72	1,985	1,936	60
8	15,900	3,999	11,901	9.57	1,954	1,906	65
9	16,320	4,234	12,086	9.71	1,983	1,934	55
10	16,200	4,209	11,991	9.64	1,969	1,920	55
11	15,815	3,759	12,056	9.69	1,979	1,930	65
12	16,310	4,309	12,001	9.65	1,971	1,922	65
13	15,900	3,999	11,901	9.57	1,954	1,906	65
14	6,500	3,950	<u>2,550</u>	<u>2.05</u>	<u>419</u>	<u>408</u>	40
			158,896	127.73	26,085	25,440	

Chronology of solution analyses from sample logbook

Experimental logbook page reference	Sample No.	Uranium concentration reported as ^{nat} U (mgU/ml)	20°C Specific gravity	Nitric acid normality	Date sample delivered for analysis
30	1A	339.1	1.4632		03-31-67
30	1B	340.2	1.4632		03-31-67
44	2	350.0	1.4838		04-10-67
62	3	353.2	1.4750		05-01-67
91	11	208.1	1.2804		06-27-67
134	12	141.6	1.1956	0.37	09-05-67
142	13	105.9	1.1867	0.50	09-14-67
150	14	133.5	1.1832	0.40	10-17-67
152	15	119.3	1.1675	0.40	10-17-67
152	16	104.7	1.1442	0.34	10-20-67
154	17	102.9	1.2117	0.36	10-20-67
158	18A	101.2	1.1392	0.36	10-24-67
158	18B	101.8	1.1441		11-14-67
170	19	85.02	1.1186		11-17-67
170	20	81.34	1.1122		11-17-67
174	21	76.17	1.1063		12-06-67
180	22	52.73	1.0746		12-06-67
188	23	45.58	1.0599		01-30-68
200	25	204.5	1.2796		02-16-68
202	26	194.5	1.2529		02-20-68
202	27	177.4	1.2357		02-20-68
210	28	134.4	1.1890		02-22-68
218	29	136.7	1.1848		03-15-68
220	30	112.6	1.1544		03-18-68
231	31	97.02	1.1401		03-27-68
238	32A	48.82	1.0683		04-01-68
238	32B	50.75	1.0658	0.24	04-19-68

A Method for Inferring the HNO₃ Concentration in a Ternary Solution of H₂O, UO₂(NO₃)₂, and HNO₃ given Temperature Dependent Solution Density and Uranium Concentration

The book entitled *Densities of Aqueous Solutions of Inorganic Substances*¹ provides partial molar volume and molar concentration relationships for determining the densities of inorganic binary and ternary solutions.

Equation (12) of the reference provides a summary equation for a temperature dependent binary solution density as:

$$\rho_s(t) = \rho_0(t) + \alpha(t)c + \beta(t)c^{3/2} \quad (12)$$

where $\rho_0(t)$ = density of water (g/l)

$$\alpha(t) = A + Bt + Ct^2$$

$$\beta(t) = -(D + Et + Ft^2)$$

resulting in equation (20) of the reference which provides the relationship for temperature dependent binary solution density as:

$$\rho(t) = \rho_0(t) + Ac + Bct + Cct^2 + Dc^{3/2}t + Ec^{3/2} + Fc^{3/2}t^2 \quad (20)$$

where:

t = temperature in °C

c = molar concentration (quantity of solute divided by volume of the solution)
(mol/l), (i.e., HNO₃ or UO₂(NO₃)₂)

A, B, C, D, E, F = constants of correlation equation

$\rho_0(t)$ = temperature dependent water density as given by reference equation (44)

$$\rho_0(t) = 999.65 + 2.0438 \times 10^{-1}t - 6.1744 \times 10^{-2}t^{3/2} \text{ (g/l)} \quad (44)$$

By mixing two binary aqueous solutions at the same temperature and with the same partial molar volume of water, the reference provides a relationship for a ternary solution with unchanged partial molar volumes of water and components. Equation (33) of the reference provides the solution density, ρ_s , as:

$$\rho_s(t) = \rho_0(t) + \sum_{i=1}^2 \alpha_i c_i - \left[\sum_{i=1}^2 \left(\beta_i^2 \right)^{1/3} c_i \right]^{3/2} \quad (33)$$

where

¹Otakar Söhnel, Petr Novotný, *Densities of Aqueous Solutions of Inorganic Substances* (Physical sciences data; 22), Research Institute of Inorganic Chemistry, Ústí nad Labem, Czechoslovakia, Elsevier, New York, New York, 1985, ISBN 0-444-99596-X.

$$\alpha_i(t) = A_i + B_i t + C_i t^2$$

$$\beta_i(t) = -(D_i + E_i t + F_i t^2)$$

for the i th solute (e.g., c_1 = molar concentration of HNO_3 and

c_2 = molar concentration of $\text{UO}_2(\text{NO}_3)_2$ in mol/l.

The constants of correlation are tabulated for HNO_3 with a molecular weight of 63.013 as:

$$\begin{array}{lll} A_1 = 4.063 \times 10^1 & C_1 = 1.096 \times 10^{-3} & E_1 = 2.478 \times 10^{-2} \\ B_1 = -1.554 \times 10^{-1} & D_1 = -2.798 \times 10^0 & F_1 = -2.761 \times 10^{-4} \end{array}$$

The constants of correlation are tabulated for $\text{UO}_2(\text{NO}_3)_2$ for a natural uranium molecular weight of 394.038 as:

$$\begin{array}{lll} A_2 = 3.182 \times 10^2 & C_2 = 2.092 \times 10^{-3} & E_2 = -1.683 \times 10^{-1} \\ B_2 = -6.041 \times 10^{-2} & D_2 = 5.374 \times 10^0 & F_2 = 1.337 \times 10^{-4} \end{array}$$

As combined in accordance with equation (33) of the reference, the temperature dependent density of a ternary solution, ρ_s , comprised of H_2O , HNO_3 , and $\text{UO}_2(\text{NO}_3)_2$ becomes:

$$\rho_s(t) = \rho_0(t) + \alpha_1 c_1 + \alpha_2 c_2 - \left[c_1 (\beta_1^2)^{1/3} + c_2 (\beta_2^2)^{1/3} \right]^{3/2} \text{ g/l.} \quad (33)$$

Because this relationship relates solution density to molar concentration of natural uranium, a correction for uranium isotopic distribution can be made for the solution density. The authors specify that the constants of correlation for the HNO_3 and H_2O solutions reproduce the experimental values at temperatures (0–100°C) and anhydrous HNO_3 concentrations (5–50 wt % anhydrous HNO_3) to within a relative standard deviation between 0.1 to 0.25. Similarly they specify that the constants of correlation for anhydrous $\text{UO}_2(\text{NO}_3)_2$ and H_2O solutions reproduce the experimental values at temperatures (0–100°C) and anhydrous $\text{UO}_2(\text{NO}_3)_2$ concentrations (5–25 wt % and 30–50 wt % at 15–25°C) to within a relative standard deviation between 0.1 to 0.25.

Given the foregoing relationships, one may infer the excess nitric acid in pure ternary solutions of H_2O , HNO_3 and $\text{UO}_2(\text{NO}_3)_2$ given the mass of uranium expressed in moles/liter and density of the solution at a given temperature. However, the known density of the predominantly ^{233}U solution must be adjusted to account for the difference in the average atomic weight of the uranium in the given solution density and the average atomic weight of natural uranium used in the development of the equations. For nearly 98 wt % ^{233}U , the mass ratio of ^{233}U to natural uranium is substantial (i.e., $233.1186/238.0289 = 0.9794$).

For illustration purposes, solution sample 11 of the report is considered. The average atomic weight of the uranium is 233.11863. The solution density is 1.2781 g/ml. The inferred uranium density from the empirical data fit shown in Figure 1 is

$$g^{233}\text{U} / \text{ml} = ((0.7413) \text{ g Soln/ml} - 0.7441)$$

$$g^{233}\text{U} / \text{ml} = 0.20338$$

$$g^{233}\text{UO}_2(\text{NO}_3)_2 / \text{ml} = \left(\frac{0.20338}{233.11863 \text{ g/M } ^{233}\text{U}} \right) \left(\frac{389.1273 \text{ g}}{\text{M } ^{233}\text{UO}_2(\text{NO}_3)_2} \right) \\ = 0.33949$$

In order to use the referenced relationship, the given density of the ^{233}U solution must be adjusted to the equivalent of a U(natural) solution (i.e., $[\text{g/M U(nat)O}_2(\text{NO}_3)_2]/[\text{g/M } ^{233}\text{UO}_2(\text{NO}_3)_2] = (394.0375)/389.1273 = 1.012619$. Therefore, the adjusted density as natural uranium solution is:

$$\rho_s = 1.2781 + 0.33949 (1.012619 - 1) \text{ g/ml} \\ = 1.2824 \text{ g/ml or } 1,282.4 \text{ g/l}$$

The $\text{UO}_2(\text{NO}_3)_2$ molar concentration is:

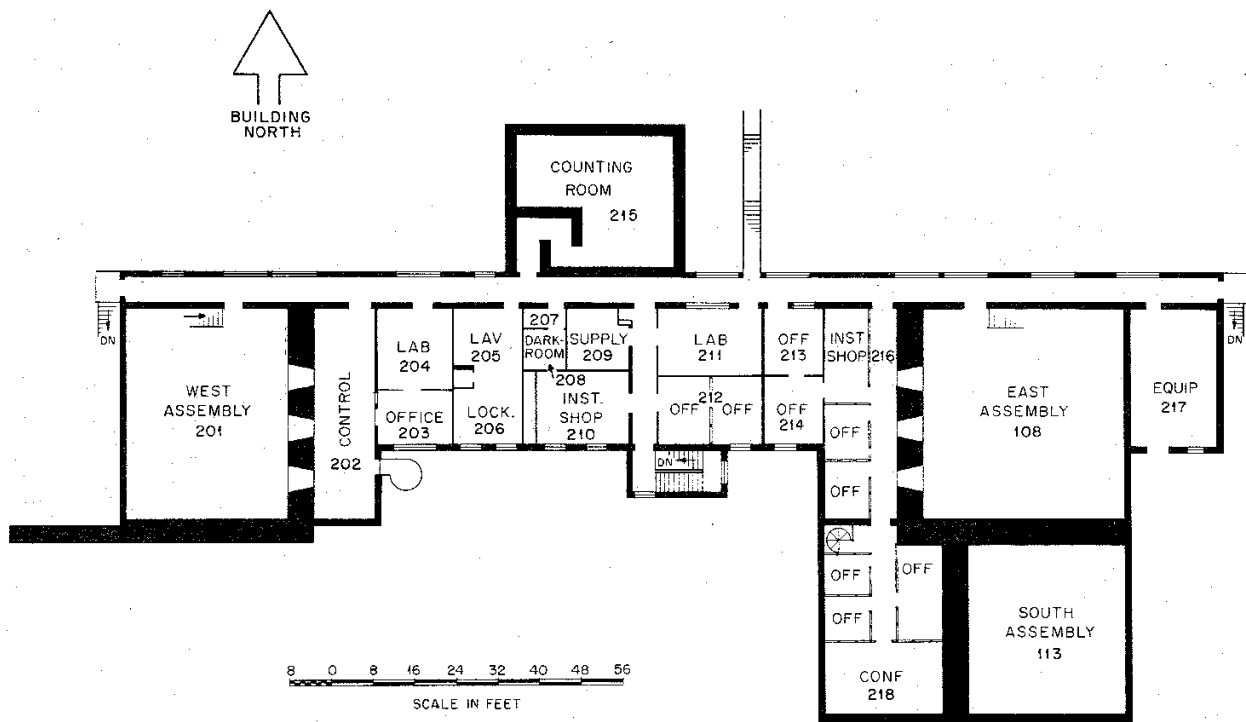
$$\left(\frac{0.20338 \text{ g } ^{233}\text{U}}{\text{ml}} \right) \left(\frac{\text{M } ^{233}\text{U}}{233.11863 \text{ g}} \right) \left(\frac{1 \times 10^3 \text{ ml}}{\text{liter}} \right) = 0.87243 \frac{\text{M } \text{UO}_2(\text{NO}_3)_2}{\text{liter}}.$$

With these two values (i.e., equivalent natural uranium solution density, $\rho_s = 1,282.4 \text{ g/l}$, and the $\text{UO}_2(\text{NO}_3)_2$ molar concentration, $c_2 = 0.87243 \text{ M } \text{UO}_2(\text{NO}_3)_2/\text{l}$, one may solve the referenced relationship for the molar concentration of HNO_3 , c_1 . The equation to be solved for the c_1 molar concentration of HNO_3 , is:

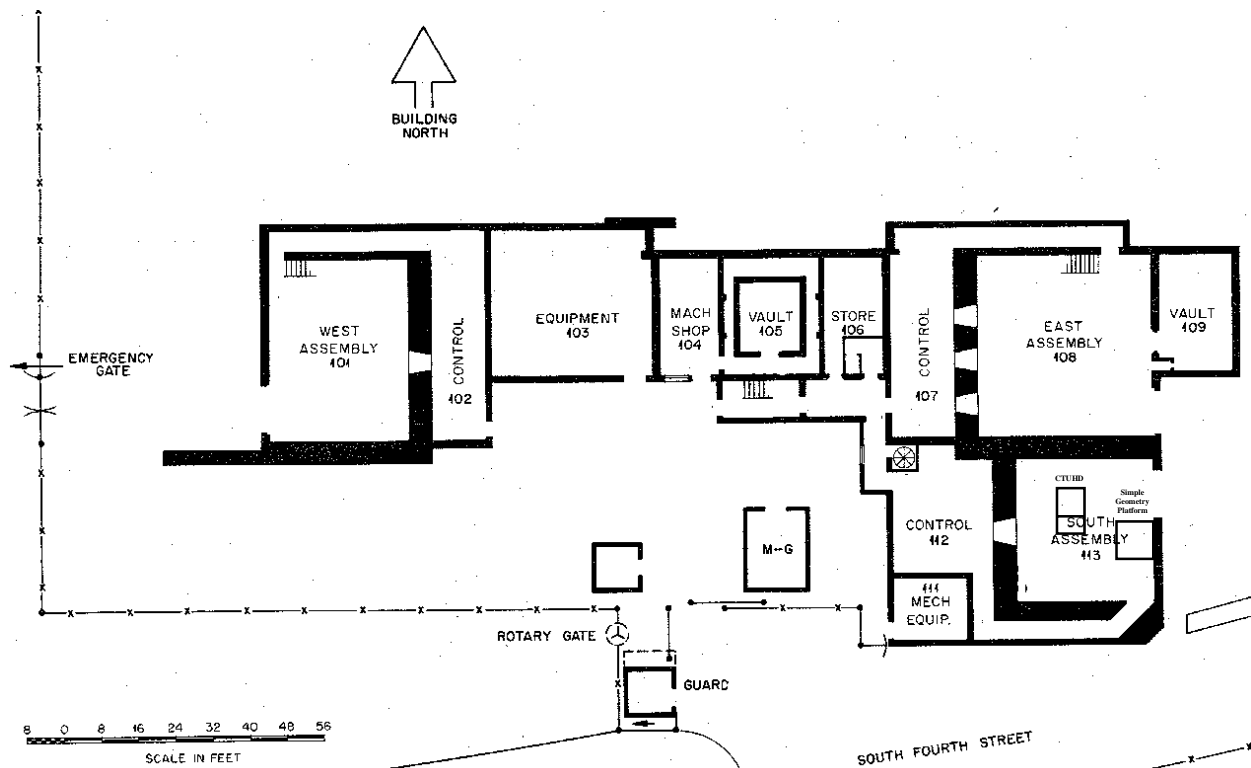
$$\rho_s(t) = \rho_0(t) + \sum_{i=1}^2 \alpha_i c_i - \left[\sum_{i=1}^2 (\beta_i^2)^{1/3} c_i \right]^{3/2} \\ \rho_s(t) = 999.65 + 2.0438 \times 10^{-1} t - 6.174 \times 10^{-2} t^{3/2} \\ + (A_1 + B_1 t + C_1 t^2) c_1 + (A_2 + B_2 t + C_2 t^2) c_2 \\ - \left\{ \left[(-D_1 - E_1 t - F_1 t^2)^2 \right]^{1/3} c_1 + \left[(-D_2 - E_2 t - F_2 t^2)^2 \right]^{1/3} c_2 \right\}^{3/2}.$$

Substituting the constants of correlation, 1,282.4 g solution per liter as $\rho_s(t)$ at the laboratory temperature, t , equal to 20°C , 0.87243 $\text{M } \text{UO}_2(\text{NO}_3)_2$ per liter as c_2 and solving for c_1 results in an HNO_3 concentration of 0.11668 $\text{M } \text{HNO}_3$ per liter. The resultant N/U atom ratio is 2.13 and H/U atom ratio is 118.6.

The foregoing illustration did not take into account the influences of impurities identified in sample No. 25, specifically the approximate 2.5 mg Th per 200 mg U in the form of $\text{Th}(\text{NO}_3)_4$. The $\text{Th}(\text{NO}_3)_4$ was present in sample No. 11. Adjustments for impurities could be approximated.

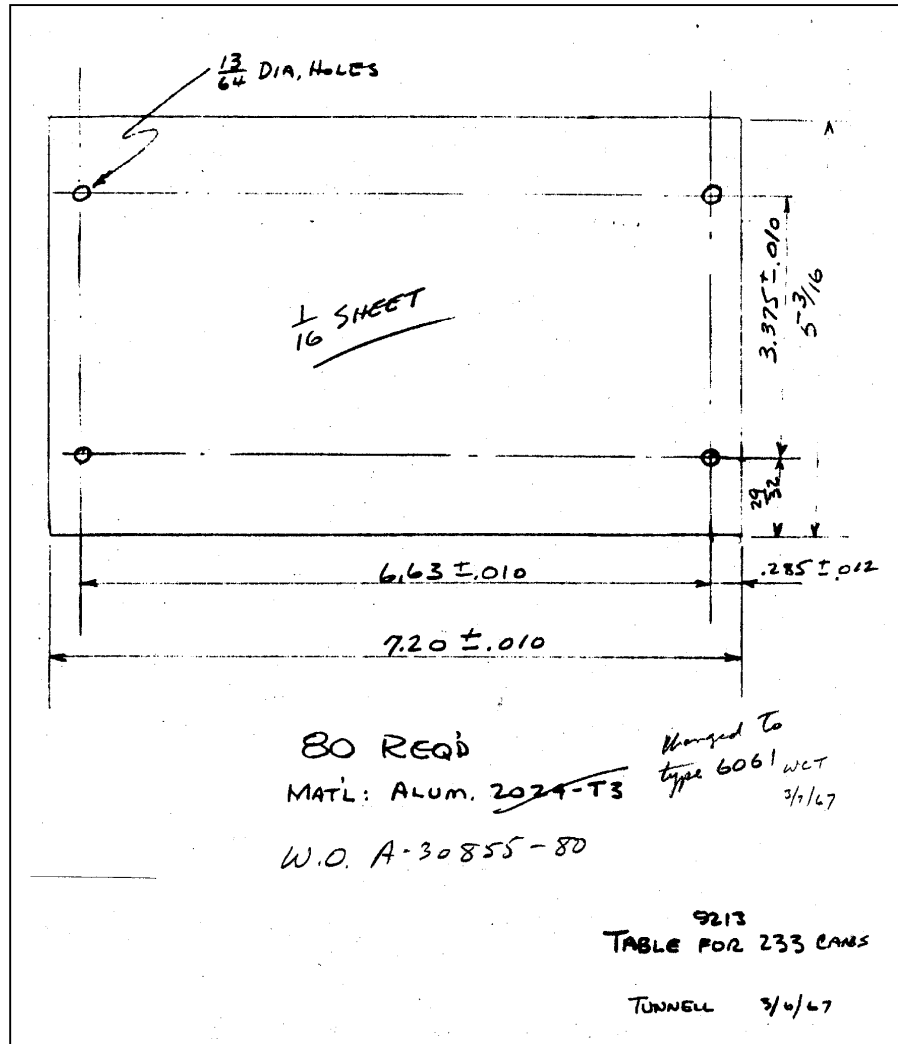


Second Floor Plan of the Oak Ridge Critical Experiments Facility

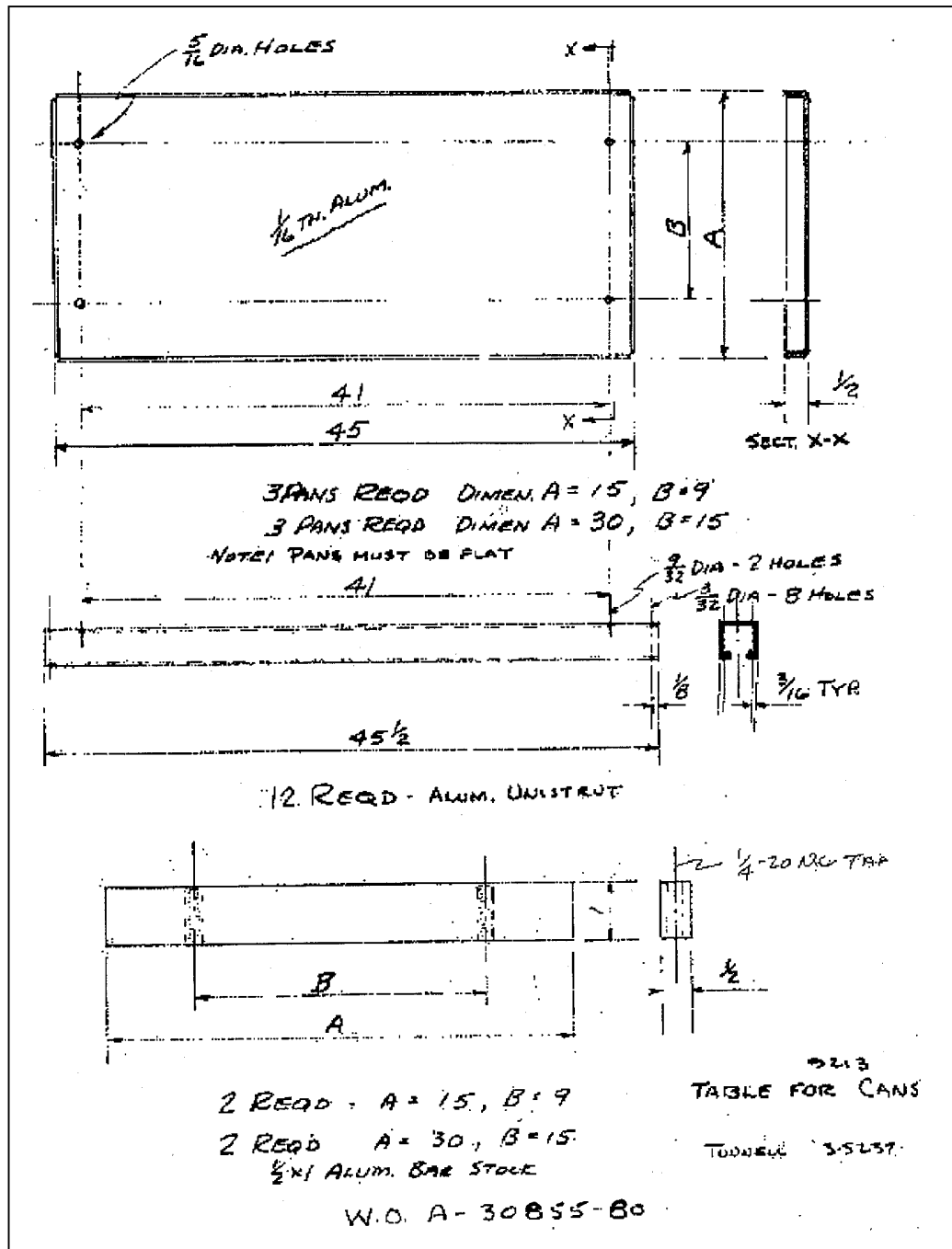


First Floor Plan of the Oak Ridge Critical Experiments Facility

Description and dimensions of aluminum plates used to support the solution containers in the unreflected array experiments.



Description and dimensions of aluminum trays used in the reflected array experiments to support the solution containers.

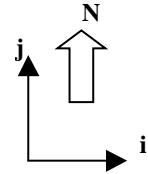


Array-cylinder identification and solution weight					
Cylinder No.	Tare wt. (kg)	Sample No. 2		Sample No. 11	
		Gross wt. (kg)	Solution wt. (kg)	Gross wt. (kg)	Solution wt. (kg)
1	0.324	6.644	6.320	5.876	5.552
2	0.323	6.643	6.320	5.877	5.554
3	0.324	6.644	6.320	5.876	5.552
4	0.323	6.643	6.320	5.877	5.553
5	0.324	6.644	6.320	5.876	5.552
6	0.325	6.645	6.320	5.878	5.553
7	0.325	6.645	6.320	5.878	5.553
8	0.323	6.643	6.320	5.875	5.552
9	0.323	6.643	6.320	5.875	5.552
10	0.324	6.644	6.320	5.865	5.541
11	0.325	6.645	6.320	5.877	5.552
12	0.324	6.644	6.320	5.876	5.552
13	0.325	6.645	6.320	5.877	5.552
14	0.325	6.644	6.319	5.877	5.552
15	0.323	6.644	6.321	5.875	5.552
16	0.324	6.644	6.320	5.876	5.552
17	0.324	6.644	6.320	5.876	5.552
18	0.324	6.644	6.320	5.876	5.552
19	0.324	6.644	6.320	5.876	5.552
20	0.326	6.646	6.320	5.878	5.552
21	0.327	6.647	6.320	5.879	5.552
22	0.324	6.644	6.320	5.877	5.553
23	0.326	6.644	6.318	5.878	5.552
24	0.323	6.643	6.320	5.875	5.552
25	0.324	6.644	6.320	5.876	5.552
26	0.322	6.642	6.320	5.874	5.552
27	0.323	6.643	6.320	5.875	5.552
average	0.32413	6.64406	6.31993	5.87596	5.55183
deviation	0.00081	0.00064	0.00021	0.00131	0.00080

CYLINDER POSITION IN ARRAY EXPERIMENTS

position (i,j,k)	Experiment Number	
	35 to 38	61 to 65
1,1,1	7	4
2,1,1	1	1
1,2,1	5	3
2,2,1	2	2
1,1,2	8	8
2,1,2	4	5
1,2,2	6	7
2,2,2	3	6

Split Table

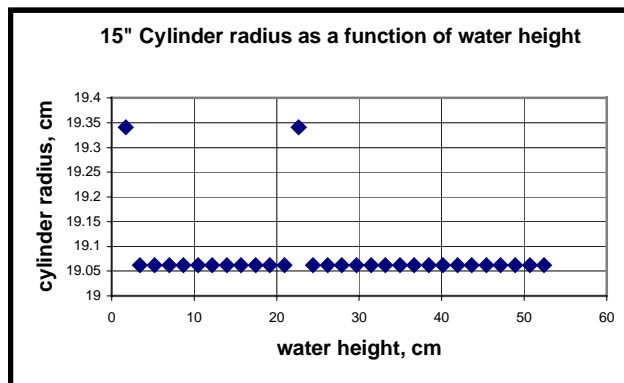


position (i,j,k)	Experiment Number						
	45	46 & 47	48 & 49	50	51 to 54	77 to 80	85 to 92
1,1,1	20	14	8	25	16	21	12
2,1,1	16	21	22	5	12	20	25
3,1,1	15	23	16	22	22	19	10
1,2,1	10	1	15	10	8	12	18
2,2,1	11	5	1	12	25	11	4
3,2,1	19	3	8	8	26	9	2
1,3,1	4	26	24	21	21	3	27
2,3,1	3	19	26	24	27	2	26
3,3,1	1	13	23	9	17	1	9
1,1,2	14	11	27	14	10	24	13
2,1,2	25	22	3	18	7	23	21
3,1,2	24	16	11	4	18	22	9
1,2,2	12	10	9	7	1	15	16
2,2,2	13	7	10	20	2	14	8
3,2,2	18	2	14	1	14	13	1
1,3,2	6	27	17	27	6	6	24
2,3,2	5	15	13	15	5	5	17
3,3,2	8	17	2	17	23	4	6
1,1,3	23	18	18	2	9	27	14
2,1,3	26	12	12	13	11	26	7
3,1,3	27	9	25	11	13	25	11
1,2,3	21	6	4	3	3	18	20
2,2,3	22	4	5	16	20	17	15
3,2,3	17	8	20	19	19	16	23
1,3,3	9	24	21	6	24	10	22
2,3,3	7	25	7	23	15	8	5
3,3,3	2	20	19	26	4	7	3

Calibrations performed by C. Cross and S. J. Rafferty during June 1968.

Water (liters)	Water height (cm)						
	15"	10"	8"				
0	0	0	0				
2	1.70	3.90	5.90				
4	3.45	7.80	12.10				
6	5.20	11.70	18.20				
8	6.95	15.70	24.37				
10	8.70	19.60	30.50				
12	10.45	23.50	36.75				
14	12.20	27.40	42.80				
16	13.95	31.40	48.95				
18	15.70	35.30	55.10	SUMMARY			
20	17.45	39.20	61.25		15"	10"	8"
22	19.20	43.15	67.40	ave. rad.	19.0625	12.7175	10.1698
24	20.95	47.10	73.55	ave. dev.	7.11E-15	0.0517	0.0333
26	22.65	51.00	79.70				
28	24.40	54.90	85.85				
30	26.15	58.90	92.00				
32	27.90	62.80	98.05				
34	29.65	66.70	104.20				
36	31.40	70.70	110.35				
38	33.15	74.60	116.50				
40	34.90	78.50	122.65				
42	36.65	82.45	128.75				
44	38.40	86.40	134.90				
46	40.15	90.30	141.00				
48	41.90	94.25	147.15				
50	43.65	98.20	153.30				
52	45.40	102.10	159.50				
54	47.15	106.00					
56	48.90	109.90					
58	50.65	113.85					
60	52.40	117.80					

Measured heights employed a 1.27-cm-ID sight glass.



GLYPTAL AIR-DRY ENAMEL (RED)

MATERIAL SAFETY DATA SHEET

SECTION I

MANUFACTURER: GLYPTAL, INC.
305 EASTERN AVE
CHELSEA, MA 02150
TELEPHONE: 617-884-6918
PRODUCT CLASS: AIR DRY ENAMEL
CODE IDENTIFICATION: 1201

TRADE NAME: GLYPTAL

HMIS 2 3 0

SECTION II - HAZARDOUS INGREDIENTS

INGREDIENT	PERCENT BY WEIGHT	PPM	ACGIH TLV mg/cu.m. PPM	OSHA PEL mg/cu.m.
XYLENE	34.5	100	100	
CAS NUMBER 1330-20-7				
HMIS HEALTH=L FLAMMABILITY=3 REACTIVITY=O				
XYLOL				
VM&P NAPHTHA	5.6	300	300	
CAS NUMBER 8030-30-6				
HMIS HEALTH=2 FLAMMABILITY=3 REACTIVITY=O				
ALIPHATIC HYDROCARBON				
STODDARD SOLVENT	0.2	100	100	
CAS NUMBER 8052-41-3				
HMIS HEALTH=2 FLAMMABILITY=2 REACTIVITY=O				
HYDROCARBON MIXTURE				
IRON OXIDE	8.2			
CAS NUMBER 1309-37-1				
HMIS HEALTH=O FLAMMABILITY=1 REACTIVITY=1				
FERRIC OXIDE				
HYDRATED MAGNESIUM SILICATE	16.9	20		
CAS NUMBER 14807-96-6				
HMIS HEALTH=2 FLAMMABILITY=O REACTIVITY=O				
TALC				

Remaining 34.7 & is a nonhazardous alkyd resin.

VM & P (CAS# 8030-30-6). AGENCY OSHA TYPE STEL EXPOSURE LIMIT 400 PPM

N/A MEANS NOT AVAILABLE N/EST MEANS NOT ESTABLISHED

NOT EST. means NOT ESTABLISHED

NOT EST. means NOT ESTABLISHED

N/A MEANS NOT AVAILABLE NOT EST MEANS NOT ESTABLISHED

SECTION III - PHYSICAL DATA

BOILING RANGE: 250.0 TO 345.0 F VAPOR DENSITY: HEAVIER THAN AIR

EVAPORATION RATE: SLOWER THAN ETHER

PERCENT VOLATILE BY VOLUME: 56.5 VOC (less water): 3.98 LBS/GALLON

WEIGHT PER GALLON: 9.91 POUNDS

V.O.C. Is determined per EPA Reference Method 24 using ASTM procedures D2369 ,D1475 and D3960.

VAPOR PRESSURE: 5.79 mm/hg MELTING POINT: NOT APPLICABLE

SOLUBILITY IN WATER: NEGLIGIBLE

APPEARANCE AND ODOR: RED LIQUID WITH PAINT ODOR

ORNL ANALYTICAL CHEMISTRY DIVISION
SPECTROCHEMICAL LABORATORY
BUILDING 9734, Y-12 TELEPHONE 3-7168

Request No **22078**

REQUEST AND REPORT OF SPECTROGRAPHIC ANALYSIS

SUBMITTED BY J. T. Thomas
COPY REPORT TO

CHARGE NO. 4410-39 SAMPLE NO. 4.2. 1500 Th
BUILDING NO. 9213 PHONE 35237 DATE 3-11-6
BUILDING NO.

COMPOSITION OF SAMPLE IF KNOWN

TYPE OF ANALYSIS DESIRED

☐ Qualitative, ☒ Semi-quantitative, ☐ Quantitative, ☐ Photoelectric

ELEMENTS DESIRED

GENERAL ANALYSIS

(values in wt %)
Alloys Thin St. T. Y. T.
Ag <0.01 <0.01 In — Sc —
Al .1 <0.2 Ir — Si ≅.5 <0.05
As — K ~0.3 <0.2 Sn <0.05 <0.05
Au — Li ~0.1 <0.05 Sr —
B .025 <0.05 Mg ≅.3 <0.1 Ta <0.2 <0.2
Ba <0.05 <0.05 Mn 0.05 <0.1 Te —
Be <0.05 <0.05 Mo <0.03 <0.02 Th —
Bi <0.05 <0.05 Na ~0.3 <0.02 Ti 0.03 <0.02
Ca ~0.2 <0.05 Ni <0.05 <0.05 Tl —
Cb <0.1 <0.1 Os — U —
Cd <0.02 <0.02 P — V ~0.03 <0.02
Co 0.03 <0.03 Pb 0.02 <0.02 W <0.2 <0.2
Cr <0.05 <0.05 Pd — Zn <0.02 <0.02
Cs — Pt — Zr <0.05 <0.05
Cu 0.01 <0.02 Rb <0.02 <0.02
Fe ~5 <0.01 Re —
Ga — Rh —
Ge — Ru —
Hf — Sb <0.2 <0.2
Hg —

RARE EARTH ANALYSIS
(values in _____)

Sc _____
Y _____
La _____
Ce _____
Pr _____
Nd _____
Sm _____
Eu _____
Gd _____
Tb _____
Dy _____
Ho _____
Er _____
Tm _____
Yb _____
Lu _____
Th _____

METAL ANALYSIS
(values in _____)

Type alloy _____
Cr _____
Ni _____
Fe _____
Mo _____
Co _____
Cb _____
Ta _____
Mn _____
V _____
Ti _____
Cu _____
Al _____
Mg _____
Sn _____
Pb _____
Zn _____
Bi _____
Si _____

Explanation of Analysis:

Symbols Used: P-Present; T-Trace; < - less than; > - greater than; nd-not detected; no analyses made in all other cases.

☐ Qualitative Analysis - Estimate only as follows: M-major; m-minor; t-trace.

☒ Semi-Quantitative Analysis - The values reported are visual estimates taken from a standard plate and using a common graphite matrix. These values are to be interpreted as approximations only. Actual value should be within the range times 1/2 to times 2.

☐ Quantitative Analysis - The values reported are obtained by visual comparison of the sample with standards similarly prepared. Precision is about $\pm 50\%$ of the amount present.

☐ Densitometric Analysis - The values reported are obtained by precise analytical spectrochemical methods. Precision of the method varies but is of the order of $\pm 10\%$ or better.

☐ Photoelectric Analysis - Rapid electronic method. Precision _____ %.

ANALYSIS PERFORMED BY SA-m Jc APPROVED BY J. A. Carter PLATE NO. 5800-B+C DATE REPORTED 3-12-68
COMMENTS

68862

TO	ANALYZED FOR
FROM	SERIES NO.

J. T. Thomas

GENERAL ANALYSIS LAB.

CONTROL NO.

CONTI

68862

[illegible]

CONCENTRATION		REMARKS	LABORATORY SUPERVISOR
		<input checked="" type="checkbox"/> SAMPLES returned to sender <input type="checkbox"/> sent to waste <input type="checkbox"/> retained	

LABORATORY SUPERVISOR

W. R. Roney

REMARKS

☒ SAMPLES returned to sender
☐ sent to waste ☐ retained

CONCENTRATION

JCN-2134
3 9-60)

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