

**BOOK25R**

10147 on bottom edge

*Notes:*

“DFC” wrote on tape attached to front

Blank pages: inside front cover opposite page 1, 2, 5-19, 27-30, 33-39, 46-49, 62, 68-152, inside back cover.

-page 23 has 8.5x11 pink sheet taped to it

-page 25 has sheet stapled to it

-page 53 has 8.5x11 sheet taped to it

*Scanned by:*

*Sheila Finch*

*RSICC /Oak Ridge National Lab.*

*August 3, 1999*

10147

SECRET

INVA  
24



SECRET

14-2-2  
Note Book #22

**SECRET**

Solution Experiments -  
Material Balance - etc

This document consists of 155 pages.  
No. 1 of 1 copies, Series A



# Standard Blank Book



No. 21

Journal	2 Cols.	to Right	Units	Single Page Form
"	3	"	"	"
"	4	"	"	"
"	5	"	"	"
"	6	"	"	"
"	6	Divided	"	"
"	8	to Right	"	Double Page Form
"	10	"	"	"
"	12	"	"	"
"	12	"	No Units	"

Single Ledger, Units  
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CLASSIFICATION CANCELLED  
DATE 5/27/60  
For the Atomic Energy Commission  
Jack H. Fisher for the  
Chief, Declassification Branch

**RESTRICTED DATA**  
This document contains restricted data as defined in the Atomic Energy Act of 1946

**SECRET**

This document consists of 155 pages.  
No. 1 of 1 copies, Series A

## Calc. of Areas:

3

Storage Cylinders:

$A_1 \approx 1. D. 3''$  Pipe (stor. cyl.)

$$A_1 = \frac{(3.068)^2}{4} \pi = 7.392 \text{ in}^2$$

$A_2 =$  Total cross sectional Area of  $\frac{1}{4}''$  Pipe

$$A_2 = \frac{(.54)^2}{4} \pi = .229 \text{ in}^2$$

$$A_3 = \text{inside A. } \frac{1}{4}'' \text{ Pipe} = \frac{(.369)^2}{4} \pi = .104 \text{ in}^2$$

$A_A =$  Area of pipe section:  $A_2 - A_3$

$$A_4 = .229 - .104 = .125 \text{ in}^2$$

To convert  $\text{in}^2$  to  $\text{cm}^2$  multiply by 6.452

$$A_1 = 47.693 \text{ cm}^2$$

$$A_2 = 1.477 \text{ in}^2$$

$$A_3 = 0.671 \text{ in}^2$$

$$A_4 = 0.806 \text{ in}^2$$

Reaction cylinder Areas:

10 inch cyl.,	$A_{10''} = 78.540 \text{ in}^2 = 506.74 \text{ cm}^2$
9 " "	$A_{9''} = 63.617 \text{ in}^2 = 410.44 \text{ cm}^2$
8 " "	$A_{8''} = 50.265 \text{ in}^2 = 324.31 \text{ cm}^2$
7 " "	$A_{7''} = 38.489 \text{ in}^2 = 248.30 \text{ cm}^2$
6 " "	$A_{6''} = 28.274 \text{ in}^2 = 182.42 \text{ cm}^2$
5 " "	$A_{5''} = 19.635 \text{ in}^2 = 126.68 \text{ cm}^2$
4 " "	$A_{4''} = 12.566 \text{ in}^2 = 81.07 \text{ cm}^2$

4

## Reaction Cylinder vs. Storage Cylinder Area

Ratios:

Net Area storage Cy). When filling Reactor  
 is  $A_1 - A_2 = 7.163 D^2 = 46.215 \text{ cm}^2$

$$\text{Ratio } 10'' \text{ Reactor} = \frac{506.74}{46.215} = 10.965$$

$$\text{Ratio } 9'' \quad " \quad = \frac{410.94}{46.215} = 8.881$$

$$\text{Ratio } 8'' \quad " \quad = \frac{324.31}{46.215} = 7.017$$

$$\text{Ratio } 7'' \quad " \quad = \frac{248.30}{46.215} = 5.373$$

$$\text{Ratio } 6'' \quad " \quad = \frac{182.42}{46.215} = 3.947$$

$$\text{Ratio } 5'' \quad " \quad = \frac{126.68}{46.215} = 2.741$$

$$\text{Ratio } 4'' \quad " \quad = \frac{81.07}{46.215} = 1.754$$

## CYLINDER TARES

*Measured F.O.T. on Ohaus Balance.*

# 1 - 13.661 Kg (Locks on)

# 2 - 13.725

# 3 - 13.824

# 4 - 13.600

5 - 13.875

6 - 13.553

7 - 14.106

8 - 13.770

9 - 13.608

(Readings 29m low  
compared to Eimer &  
Amend Wts.)

Three weights, at 4-12, of filled cylinders -

	8/28/47 Measured at 4-12 on <del>16.6788</del> Kg.	F.O.S. Actual Balance - → Nil wts ← using F.O.S. wt.	8/28/47 at F.O.S. gross wt.
*1	16.6788	3.018 Kg    3.017	16.678
*2	16.8488	3.124 Kg    3.123	16.848
*3	16.9348	<del>3.111</del> 3.111    3.109	8/29 at F.O.S. 16.933
*4	16.6515	3.052    3.051	16.651
*5	17.0352	3.160    3.160	17.035
*6	16.6069	3.054    3.055 <del>3.0578</del>	16.6098
*7	17.8554	3.749    3.751	17.857
*8	17.6790	3.909    3.912	17.682
*9	16.7233	<u>3.115</u> 29.292	3.113    16.721
			<u>29.291</u> ✓ total wt.

12/1/48  
X

29.292 x 0.4641 = 13.594 kg U (analysis on following page)  
13.                    = 12.697 kg X

22

Samples taken from Cylinders 1-9 etc. for Run 8 Lab D.  
8/29/47

Net wts

Parlett - Callahan

#1

#2

#3

Cylinder #1

~~2.4370~~

2.3749

0.6880 gm.

0.5661 gm

Cylinder #2

0.5165

0.5128

Cylinder #3

0.7574

0.6885

1.3565

Cylinder #4

0.6749

0.7801

Cylinder #5

0.9310

0.6534

Cylinder #6

0.3977

0.5678

Cylinder #7

0.6094

0.6221

Cylinder #8

0.6726

0.6402

Cylinder #9

0.5907

0.5679

1.6622

Total 16.8307 gm

7.8106 gm T



TRANSFER OF URANIUM MATERIAL  
CARBIDE AND CARBON CHEMICALS CORPORATION  
K-25 PLANT

ACCOUNT CODES

96	65
----	----

C-2870

1

FROM: V-8A

MATERIAL CODE FROM TO TRANSFER NUMBER

TO F-05

DATE 8-29-47 TIME 2: AM

DATE 8-29 TIME 3:10 PM

SHIPPERS' SIGNATURE *J. K. ...* LEAVING SENDER

RECEIVER'S SIGNATURE *Al ...* RECEIVED

MATERIAL DESCRIPTION C-2870

DOCUMENT CLASSIFICATION F.R.

CONTAINER NO.	OPERATOR'S GROSS	OFFICIAL GROSS	OFFICIAL TARE	OFFICIAL NET	SAMPLE REQ. NO.				
1	16678	16679	13661	3018					
2	16848	16849	13725	3124					
3	16933	16935	13824	3111					
4	16651	16652	13600	3052					
5	17035	17035	13875	3160					
6	16608	16607	13553	3054					
7	17857	17855	14106	3749					
8	17682	17679	13770	3909					
9	16721	16723	13608	3115					
TOTALS									

UNITS OF MEASUREMENT: CIRCLE ONE: Pounds Kilograms Grams Liters Gallons Pints

DISTRIBUTION: White Copy - Coded Chemicals K-303-B; Canary Copy - Sender; Pink Copy - Receiver.

WCX-703 (AUG 47)

SENDER RESPONSIBLE FOR SIGNATURES AND DISTRIBUTION

103.37 gm net.

IX - 10

~~Exp. #~~  
K-25 Analyses:

TOTAL T  
46.60%

ASSAY % X

46.49

AU. of Three  
samples:

92.91 ± 0.28%

46.56

4/14/49 Flanders reports records  
done 9/17/47

46.65

Cylinder 1	90.39
3	92.44
9	91.76

46.13

Rerun on ~ 9/26/47

# 1	92.95
# 3	92.96
# 9	92.85

Probably after purification  
of samples -

46.08

46.76

46.07

46.33

AU. 46.907% T

↘  $\frac{H}{X} = 24.0$

(43.3% H<sub>2</sub>O)

WCS-708 (Aug 47)

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## Y-12 Data

<del>72</del>	T Analyses <del>72</del>		AU. ASSAY	Kg. X	
	<u>90</u>	<u>Kg T</u>			
#1	46.58	1.406	934	1.313	1313
#2	46.60	1.456	934	1.360	1360
#3	46.39	1.443	934	1.348	1348
#4	46.67	1.424	934	1.330	1330
#5	46.20	1.460	935	1.364	1365
#6	45.97	1.404	935	1.311	1313
#7	46.64	1.750	934	<sup>63</sup> 1.364	1635
#8	46.13	1.803	935	1.684	1684
#9	46.31	1.493	934	1.348	1348

Total 13.589 Kg T

12.692 Kg X

12681  
12699

ave 46.39%

# Material Charge

✓	Cylinder	1	2
✓	Gross	16,678.8	16,84
✓	Tare	13,661.	13,72.
✓	Net	3,018.	3,12
	Batch #	11-7821	11-781
		11-7848	11-784
		11-7798	11-779
		11-7801	11-780
	K-25 →	.4660	.9649
✓	T 9/9	.4658	.4660
	T	1,406.	1,45.6
	X	1,313.	1,360
	NH/Nx	<del>32.07</del>	<del>32.04</del>
		23.53	23.51
✓	Reg. #	808440	808439
	Assay	935	935
		934	934
		934	934
		933	933
✓	Ave	934	934

d to F-05

	3	4	5	6	7	8	9	tal
16,9348	16,6515	17,0352	16,6069	17,8554	17,6790	16,7233		
13,824	13,600	13,875	13,553	14,106	13,770	13,608		
3,111	3,052	3,160	3,054	3,749	3,909	3,115		29,292
	✓	✓	✓	✓	✓			
11-7808	11-7804	11-7846	11-7841	11-7739	11-7826	11-7812		
11-7810	11-7819	11-7832	11-7855	11-7853	11-7828	11-7806		
11-7833	11-7802	11-7842	11-7830	11-7850	11-7791	11-7824		
11-7835	11-7817	11-7818	11-7823	11-7839	11-7837	11-7799		
				11-7851	11-7857			
.4656	.4665	.4613	.4608	.4676	.4607	.4633		
.4639	.4667	.4620	.4597	.4669	.4613	.4631		ditto
1,443	1,424	1,460	1,404	1,750	1,803	1,443		13,589
1.348	1.330	1.364	1.311	1.634	1.684	1.348		12,692
<del>32.31</del>	<del>31.95</del>	<del>32.56</del>	<del>32.86</del>	<del>31.72</del>	<del>32.65</del>	<del>32.42</del>		
23.78	23.42	24.03	24.33	23.70	24.12	23.89	23.78 AU	
808438	808437	808436	808435	808434	808433	808432		
931	935	935	935	932	935	934		
934	933	936	935	935	936	933		
935	933	935	936	936	935	935		
935	933	934	935	935	935	934		
		935		934	936			
934	934	934	935	934	935	934		

# Material Charged to F-05

	Y-12 Data	K-25 Data
Total T	<u>13,589 Kg</u>	<u>13,593 Kg</u>
" X	12,692	12,631 <sup>X0.9292</sup> <del>kg</del>

*[Faint, illegible handwritten notes at the bottom of the page, possibly bleed-through from the reverse side.]*

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10/8/47

Sample taken from cyl #3 after dilution to density 1.47 in  $\frac{1}{4}$  60

net wt.

dish #36

4.5445 gms

## Inventory of Evaporated Solution

(correction for paper on  
cylinder = 70 gm)

Cylinder #3  
(into #3 in pot)  $\begin{array}{r} \text{gr.} \\ \text{tare} \\ \hline \text{net} \end{array} \begin{array}{r} 15.970 \\ 13.820 \\ \hline 2.150 \end{array} \approx 1.119 \text{ l} \approx 858 \text{ gm} \times$

sp. gr.  $1.93 \approx 29.5\% \approx 770 \text{ gm/l}$ 

Cylinder #4 (Full - too heavy for balance) - 50# on scale  
(into #2 in pot) Contains 4.25 liter  $\approx 3485 \text{ gm} \times$   
sp. gr.  $2.00 \approx 26.5\% \approx 820 \text{ gm/l} \times$

Cylinder #5  
(into #1 in pot)  $\begin{array}{r} \text{gr.} \\ \text{tare} \\ \hline \text{net} \end{array} \begin{array}{r} 18.030 \\ 13.875 \\ \hline 4.155 \end{array} \text{ gm} \approx 1.997 \text{ l} \approx 1757 \text{ gm} \times$   
sp. gr.  $2.08 \approx 29\% \approx 880 \text{ gm/l} \times$

Cylinder #7 (Full - 50# on large scales)  
(into #1 in pot) Contains 4.25 liter  $\approx 3443 \text{ gm} \times$   
sp. gr.  $1.98 \approx 27\% \approx 810 \text{ gm/l} \times$

Cylinder #8  
(into #2 in pot)  $\begin{array}{r} \text{gr.} \\ \text{tare} \\ \hline \text{net} \end{array} \begin{array}{r} 16.463 \\ 13.770 \\ \hline 2.693 \end{array} \text{ gm} \approx 1.367 \text{ l.} \approx 1073 \text{ gm} \times$   
sp. gr.  $1.97 \approx 27\% \approx 810 \text{ gm/l} \times$

Cylinder #4

Total 12.978 l.  $\approx 10,616 \text{ gm} \times$



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ROUGH SOL. INVENTORY

PIT.	Cyl. #1	2	3	5	6	7	8
liters	3.48	3.80	5.62	6.34	5.98	6.46	6.12
Kg Sol.	3.98	4.34	6.43	7.23	6.84	7.39	7.00
Kg X	0.405	0.440	0.653	0.734	0.694	0.750	0.710
Total X		4.386					4.386

# Samples Sent To Lab. During Solution Experiments

(Weights etc. entered in sol. Expt Book)

(See also pp 22-23 for analysis of solutions as received from 4-12).  
 $H/X = \frac{2797}{11}$  - 36.30 when  $V = 9.0$  in 43.4%  $UO_2F_2 + H_2O$

Approx. H/X	Dish NO.	Cylinder NO.	Sample Wt. (Net) (solution unless otherwise noted)	% U wt. = W	WT U	Actual H/X	Sent to	Date Samples
30	25	9	1.2082	41.83	.51	31.0	Anal Sent	9/10/47
"	34	5	0.9442	41.30	.39			9/10/47
37	35	6	2.1388	37.45	.80	38.1	"	9/16/47
"	37	8	2.4103	37.69	.91			9/16/47
45	<del>31</del>	9	2.8229	33.89	.96	46.2	"	9/17/47
"	36	7	3.1818	33.89	1.08			9/17/47
66	32	6	1.6201	28.79	.47	61.2	"	9/22/47
"	33	6 (Dup)	2.2527	28.72	.65			9/22/47
"	19	2	2.8187	28.71	.81			7/22/47
"	29	2 (Dup)	1.7393	28.77	.50			9/24/47
"	36	#2,5,7	4.5445	28.8	1.32			10/8/47
	<del>32, 27</del>	Composite	3.0480	28.8	-	-	Optical Spec	10/21/47
	27	Composite	0.25 gm $U_3O_8$	-	0.21	-	Optical Spec	10/21/47
	27	"	-	-	0.67	-	Work Lab	10/21/47
	37	Purified oxide	0.25 gm $U_3O_8$	-	0.21	-	Optical Spec	10/21/47
	41 + 37	"	1.72 gm $U_3O_8$	-	1.46	-	Work Lab	10/21/47
	<del>31</del>	Composite Evap. sol.	1.50 gm $U_3O_8$	-	1.27	-	Opt. Spec	10/24/47
	37	Purif. Oxide	0.7657 gm $U_3O_8$	-	0.648	-	Opt. Spec	10/24/47
Total to date					(12.87)			
	# 26	Solution	0.4217 gm $U_3O_8$		0.356		opt spec	11/7/47
	30	Residue from Ni wrap pan	0.3736 "		0.316		opt spec	"/10
	L-6	Recovered mat	0.1841 gm $U_3O_8$				opt spec	"/14
	35	Soln	1.5217 gm $U_3O_8$		1.29		Lab	12/12
	<del>24</del>	Soln	1.0854		0.918		"	"
	38	Soln	0.8897		0.753		opt. spec	"
	30	purif oxide	0.4127		0.095		opt spec	"
Material returned from lab			" "		0.403		opt spec	Rec'd Dec 1/47
Returned from Lab. 12/30/47			0.7443 gm $U_3O_8$		0.6384			

See page 42 for further samples.

$\frac{U_3}{O_{308}} = \frac{705}{833} = 0.8463$

Record

Report

Correct

H<sub>2</sub>

H<sub>2</sub>

H<sub>2</sub>

H<sub>2</sub> { also run on optical spec.

Attached

Corrosion ex.

Sartip 2  
Part of recovery procedure  
Sartip 2

Samples not reported - no results obtained  
93.26 ± 0.11  
Samples not reported - no results obtained  
93.29 ± 0.13 (#3) 93.10 ± 0.22

contaminants  
"

Attached  
Attached

S Contaminant  
V. Contaminant  
for B + S

for ~~to be~~ Fe 14% U  
duplicate

1723 Reported 34.98% U by wt in solution  
1723 " 34.99% U " " " "

Ni, Cr, Fe for Corrosion checks  
contaminations

Date	Dish No.	Wgt. Orid	Wgt. X	Nature	To.	Remarks
1/9/48	40	1.2302	<del>0.9724</del> 0.9724	Composite	Optical spec.	For corrosion check
1/9/48	26	.6009	0.4750	Composite in duplicates	Analytical for % U.	
"	26	.5844	0.4619			Returned from Opt. Spec.
1/21/48	40	0.9842	0.7779	Returned sample		
Net shipped in Jan.		0.2116 gm. U.	0.1672 gm.	Not included in material balance because less than 1 gm.		
2/4/48	40	0.7578		Composite	Optical spec.	for corrosion check
	L1	0.1335		from Net (1/14) 50% U.	Optical spec.	for Fe, Cu etc.
	38	5.04659		from		
	27	4.6785		extracted	Analytical	for % U.
	28	7.1327		refined		
	25	0.6816		purified	for % U.	
	25	0.7949		optical duplicates		
2/17/48	G.S. bottle					
	Gross	891.841				
	Tare	443.273				
	Net	448.568	353.563	Purified Oxide	Asst Chem.	% of theoretical = 99.71% 84.40% U
		378.591 gm U				
3/12/48	30	0.1253		sample	anal. of	
	31	0.1018		of oxide		
	37	0.0725		"	Spec.	
	Bottle					
	Gross	715.382				
	Tare	315.446				
	Net	399.936				
		332.6 gm U.				
} This was re-purified & shipped left -						

Returned  
wgt X

0.7779

44

## Material Shipped

Date	Nature	Wgt.	% X	To
5/8	opide stomach (P-2)	.9950		Lab D
	" (P-3)	1.0170		Lab D

5/20	Solution #156	50 ml 7.24.1044	.0353 gm/gm	Work's lab
	#157-8	1.044	.0353 gm/gm	"

Date	Soln	F-#	50 ml	% X	Wgt.	% X	Total X
5/4		F-2	50 ml	1.35	.006 gm/gm		Work's lab
		F-3		1.24	.0092		
		F-4		1.32	.0115		
		F-5		1.27	.0145		
		F-6		1.36	.0088		
		F-7		1.37	.0066		
		S-2		1.27	.00539		
		S-3		1.28	.0023		
		F-8		1.38	.0046		
		F-9		1.39	.0121		

Total gms U = 5.347  
 Total gms X = 4.99 gms } Sent as samples

5/20	C-1	0.13 gm/site
	S-4	3.76 " "
	S-5	1.05 " "
	F-10	8.36 " "

6/17/48

45

Sample	Vol.	Sr %	% U	% X
S-6	100	1.29		
S-7	"	1.24		
S-8	"	1.25		
S-9	"	1.18		

} to wash lab

total

### Purified Oxide

P-3

	709.850		
	315.146		
Net	<u>384.404</u>	80.07% U	to Cooled Chem.
	344.406		
	307.792 gm U		

P-3A

	735.885	83.43% U	to Cooled Chem.
	275.205		
Net	<u>460.480</u>		
	384.178 gm U		

total oxide shipped to date = 1,198,447 g

previous 378,591 gm U

1,070,561 gm U

999,909 gm X

Sample from 1<sup>st</sup> Evap. Batch for #/X

$$\begin{array}{r} \text{UO}_2\text{F}_2 \\ \text{sol} \end{array} \quad \begin{array}{r} 8.5183 \\ 6.5010 \\ \hline 2.0173 \end{array} \quad \text{L-5}^-$$

$$\begin{array}{r} \text{U}_3\text{O}_8 \\ \hline 7.6641 \\ 6.5010 \\ \hline 1.1631 \\ \times .8462 \\ \hline .9849 \text{ gm U} \end{array}$$

$$\frac{\text{U}_2}{\text{U}_3\text{O}_8} = .8462$$

$$\frac{.984}{2.0173} = 98.80\% \text{ U}, \quad 1.298 \times 4880 = 6340\% \text{ UO}_2\text{F}_2$$

$$= \frac{4555}{235} X = .1938 \text{ mole} \times 100 \text{ gm}$$

$$\frac{\#}{X} = \frac{2 \text{ MOF}}{\text{AMW}}, \quad \frac{\frac{\text{MU}}{\text{MOF}}}{\frac{\text{MU}}{\text{MW}}}$$

$$\begin{array}{r} 100.00 \\ 63.40 \\ \hline 36.60 \text{ H}_2\text{O} \\ \hline 9 \\ \hline = 4.167 \frac{\text{mole}}{100 \text{ gm}} \end{array}$$

$$\frac{\#}{X} = \frac{4.167}{.1938} = 22.0$$



Calibration of P-10 pipette: 10-17-51

Wt H<sub>2</sub>O to  
Mark

$$\begin{array}{r} \#1) 10.3817 \\ \underline{6.8597} \\ 3.5220 \end{array}$$

L-4  
Dish.

$$\begin{array}{r} \#2) 10.3917 \\ \underline{6.8597} \\ 3.5320 \end{array}$$

AV. 3.527 gm H<sub>2</sub>O

SP. GR. of 1<sup>st</sup> Evap. Batch.

$$\begin{array}{r} L-4 \quad 14.3457 \\ \underline{6.8597} \\ 7.4860 \end{array}$$

$$\frac{7.486}{3.527} = 2.14$$

of 1<sup>st</sup> 2<sup>nd</sup> Evap. Batch 10-20-47

$$\begin{array}{r} 13.8938 \\ \underline{6.4783} \\ 7.4155 \end{array} / 3.527 = 2.10$$

$$\begin{array}{r} H/X \quad 10.6350 \\ \underline{6.4783} \\ 4.1567 \text{ gm O}_3\text{O}_8 \\ \underline{.8942} \\ 83134 \\ \underline{249402} \\ 166268 \\ \underline{332536} \\ 3.51739954 \\ 47.9360 \end{array}$$

$$\begin{array}{r} H \\ X = \frac{2797}{12} = 30.3 \\ \\ = 22.67 \end{array}$$

52 U<sub>3</sub>O<sub>8</sub> Sampler For Corrosion Test!

(10)-10-47

H?

Dish # 26

9.2696  
8.8484  
-  
.4212 net

General System  
Corrosion  
sent to lab. 10-10-47

Dish # 30

9.4524  
9.0788  
-  
.3736 net

Corrosion of  
nickel pan  
sent to lab 10-10-47

probably

Sampler for H/x

(10)-11-97 (2nd dilution series)

H/x = 30

Dish # 28  
Cyl. # 1

10.8665  
8.5062  
-  
2.3603

Dish 27  
Cyl 9

11.3078  
8.7344  
-  
2.5734

U<sub>3</sub>O<sub>8</sub>

9.6571  
8.5062  
-  
1.1509

9.9539  
8.7344  
-  
1.2195

H/x

31.42

33.38

Department Analytical

Department Supervisor E. Olszewski

Copy No. \_\_\_\_\_

SAMPLE	U Salts	Description	Sequence No.	Date Rec'd.	Date Finished
		I6 and #26		1-9-48	1-15-48

RESULTS	I6	U .5108 g.	$\frac{2.9675}{.5108} = 13.60\%$	X	$\frac{126.03}{13.60} = 9.2$
	26	U .4978 g.	$\frac{2.8758}{.4978} = 13.68\%$	X	$\frac{125.57}{13.68} = 9.18$

Date Reported 1-15-48

Operator Marshall

Data Book No. HL 737

Page No. 143

Supervisor Shorten

REMARKS: *These samples contained iron, and since analysis was run so as to include iron these figures for wt of U may be slightly high. (etc)*

Work Requested and Reasons for Submission of Sample: Analyses of #16 and #26 for total Uranium 235.

Date Submitted: 1-9-48

Submitted By: Cronin per E Callihan

Source of Sample Lab. B

SAMPLE HISTORY \_\_\_\_\_

Samples for HX ratios

11/2 10/21/47

# 28  
 $\frac{10.3171}{8.5061}$   
 1.8110 gms sol

# 27  
 $\frac{10.1596}{8.7343}$   
 1.4253 gms.

4141  
 4102  
 4120  
 0.8861 gms opial  
 .7499 g T  
 calc HX = 31.12

0.6909 gms anal  
 .5897 g T  
 31.82

27.07

31.56

17/11/47 Measurement of HX = 43.87  
 Sp Gr = 1.667

17/2/47 Measurement of HX = 62.6  
 Sp Gr = 1.501

17/3/47 HX = 86.3  
 Sp Gr = 1.353

1/8/47 Measurement of HX approx 125  
 Sp Gr. = 1.250

L6 2.9675 gms sol.  
 .6009 gms opial  
 HX = 128.74

26 2.8758 gms sol.  
 .5844 gms anal  
 HX = 126.16

avg = ~~127.45~~  
 126.40

from lab. 126.03

125.16

avg = 125.57

1/24/48

$d = 1.1864$   $H_x \approx 175$

Weight Burette

96.8783

Dish # L2 6.4689 gm

" L3 6.4779 gm

Wgt. Burette - L2 88.9175

Wgt. Burette - L2 & L3 81.5562

Wgt. # L5 <sup>71816</sup> 6.5003

Wgt. L5 7.1816

+ sample           
                  .6816

Wgt #25 8.9956

9.7405

0.7949

L3 7.6337

6.4779

wgt. Open           
                  1.1558

88.9175

81.5562

wgt. soln  $\rightarrow$  7.3613

wgt T = .9778

g/g  $\text{Cu}_2\text{S} = .1328$

L-2 7.7189

6.4689

wgt. Open  $\rightarrow$  1.2500

96.8783

88.9175

wgt. soln  $\rightarrow$  7.9608

wgt. T = 1.058

g/g  $\text{Cu}_2\text{S} = .1329$

~~13.29%~~  $\text{Cu}_2\text{S}$

~~6.21~~ 13.29%  $\text{Cu}_2\text{S}$

12.4190 %

13.29%  $\text{Cu}_2\text{S}$

12.4190 %

$\frac{N_H}{N_X} = 173.96$

$N_H$

$\frac{N_H}{N_X} = 173.95$

$N_H$

188

check  
70  
mainly  
and  
representative

1/30/48

dish # 38 15.2254 9.1267  
 " 8.2280 8.2240  
 sol'n → 7.0014 gms 0.9027 gms oxide

Sp. Gr. = 1.149  
 NH/N<sub>x</sub> = 219.72

2/4/48 H/X n 250

①\* dish # 40 9.8415 8.7837 gms  
 NH/N<sub>x</sub> = 225.63  
 wgt sol'n 6.0126 g 0.7578 gms oxide = 9.96% X  
 dish # 41 9.5355 8.8260  
 0.7095 gms oxide

Sp. Gr. = 1.145  
 NH/N<sub>x</sub> = ~~225.76~~ 225.76  
 wgt burette 97.3100  
 91.2974  
 6.0126

②\* wgt sol'n 5.6350 g = 9.95% X  
 NH/N<sub>x</sub> = 225.89

② 85.6624  
 5.6350

dish # 32 13.2100 8.2235  
 sol'n 5.0465

2/12/48 H/X n 325

dish # L-3 7.4709 6.4790  
 wgt. Oxid = .9919

dish # L-2 7.4942 6.4695  
 wgt. Oxid = 1.0247

wgt burette = 104.7333  
 94.0248  
 gms sample = 10.7085

wgt burette = 94.0248  
 82.9723  
 gms sample = 11.0525

sp. Gr. = 1.103

NH/N<sub>x</sub> = 320.11

NH/N<sub>x</sub> = 319.77

avg NH/N<sub>x</sub> = 319.94

% X = 7.32

% X = 7.33

avg NH/N<sub>x</sub> = 7.32%

Checked  
11/25/50

\* 11/26/48 DC: The values of 9.96% & 9.95% X do not follow from masses of pure U<sub>3</sub>O<sub>8</sub>; they are probably based also on some analysis, of which we find no record, showing impurity. These values (9.96 & 9.95%) are being assumed correct.

56

#40

$$\begin{array}{r} 9.6968 \\ 8.7840 \\ \hline \text{wgt solids } .9128 \end{array}$$

$$\begin{array}{r} 112.7672 \\ 103.4462 \\ \hline \text{wgt sol'n } 9.3210 \end{array}$$

#41

$$\begin{array}{r} 97.021 \\ 8.8265 \\ \hline \text{wgt solids } .8956 \end{array}$$

$$\begin{array}{r} 103.4462 \\ 94.5087 \\ \hline \text{wgt sol'n } 8.9375 \end{array}$$

Specific Gr. = 1.110 @

NH<sub>4</sub>/N ≈ 370

300.80

283.13

2/28/48

$$\begin{array}{r} 9.9610 \\ 9.9610 \text{ Exp 121} \\ 9.0789 \\ \hline .8821 \end{array}$$

L3

$$\begin{array}{r} 7.0529 \\ 7.0530 \\ 6.4788 \\ \hline .5742 \end{array}$$

NH<sub>4</sub> = 299.61

287.57

$$\begin{array}{r} 110.5691 \\ 88 \\ \hline 101.5932 \end{array}$$

Sp. Gr. = 1.108

$$\begin{array}{r} 101.5932 \\ 95.7405 \\ \hline 5.8527 \end{array}$$

$$\begin{array}{r} \text{wgt sol'n } 8.9758 \end{array}$$

Exp 122

$$\begin{array}{r} 7.0213 \\ 7.0213 \\ 6.4698 \\ \hline .5515 \end{array}$$

#44

$$\begin{array}{r} 7.5209 \\ 7.5301 \\ 6.8601 \\ \hline .6700 \end{array}$$

$$\begin{array}{r} 101.9120 \\ 97.2820 \\ \hline \text{wgt sol'n } 4.6300 \end{array}$$

Sp. Gr. 1.108

$$\begin{array}{r} 97.2820 \\ 89.4557 \\ \hline 7.8263 \end{array}$$

240.95

246.32

Send to Room 8

for analysis

3/5/48

10/26/49 - These materials probably returned to FOJ by Lab after analysis - DC.

wgt. burette class = 75.3443 gm

3/5/48 By Diphone from lab.

Samples sent to Rem 8 in wgt. burette

Ex.	Wt. (gm/cc)
121	.09113 gm/cc
122	.09136 gm/cc
123	.26574 gm/cc
124	.26947 gm/cc

These samples were pipetted out and material electrolyzed to remove U  
20.27 g/g U

H/X's calculated using Sp. Gr. as det'd at 7.05

Ex.	Sp. Gr.	H/X	U %
121	1.108	281.83	
122	1.108	280.15	
123	<del>1.311</del>	92.43	101.66 - 18.93% U
124	1.317 (?)	91.24	100.45 20.27
125	1.319 (1)		24.80% U
126			20.88% U

(Sp. Gr. was assumed after 20.96 conform)

20.60% U av. of 123-126  
99.45 H/X av. of 123-126



58

3/8/48

26-125

30 ml sp. Gr. 1.319

3/22/48

Samples of purified Oxide

#30	9.2041
	<u>9.0788</u>
	0.1253

#31	8.2526
	<u>8.1408</u>
	.1018

lab analysis = 81.05%

lab analysis = ~~81.11%~~ 81.11%

95.8% U<sub>2</sub>O<sub>8</sub>

#37	8.9094
	<u>8.8369</u>
	.0725

8-12-48 Sample of Refined Oxide

16.4556
<u>14.0770</u>
2.3786

Bottle of refined Oxide (sample above)

55 <sup>2</sup> <u>093</u>
282.008
<u>270.085</u>

6010-20-48 After addition of  
12. Jap. samples to refined oxide

	570.535
	282.008
P-5	288.527

6-29-49

61

NORMAL UF<sub>4</sub> Questionable Purity

842.521  
 449.824  
 -----  
 392.697  
 207

N-1

% UF<sub>4</sub>  
 75.8

~~Sample 19.715~~

NORMAL UF<sub>4</sub> (Presumed to be fairly pure)

715.980 tare  
 376.830 tare  
 -----  
 399.150 net (gms)  
 5.237 sample  
 -----  
 393.913 ✓

N-2

24.952 gm  
 19.715  
 -----  
 5.237  
 Sample

IMPURE NORMAL UF<sub>4</sub> + PTFE

~ 86.3 wt. % UF<sub>4</sub>  
 13.7 " " " PTFE

899.292 m.  
 343.619 tare  
 -----  
 555.673 gm net  
 6.514 sample  
 -----  
 549.159

(Probably 10 gm  
 impurity. H<sub>2</sub>O)

N-3

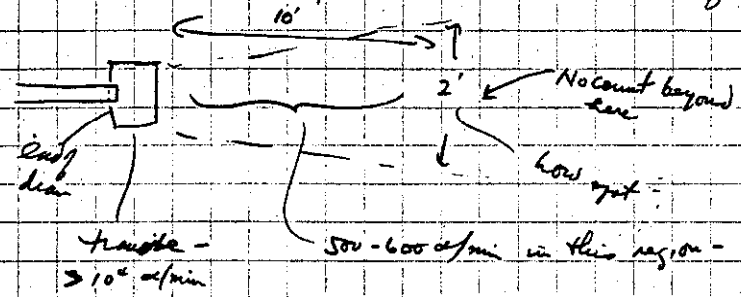
27.182 gm  
 20.668  
 -----  
 6.514  
 Sample

9/6/49

For  
CLONIX  
CALLINAN

Discovered evidence of leak in S'al reactor:

- ① High count in bottom of sample tank - visible
- ② " " " " bottom edge of al water can.
- ③ Sample at exit of 3" sample drain line -  
10' from exit - 500-600  $\alpha$ /min on gravel.  
Several thousand  $\alpha$ /min on flange at end of pipe



- ④ Stainless steel al
- ④ 6' water used in sample tank Exp 261, 8/30 - no observed change in night glass rdg, during exp.

⑤ Stainless steel shells, used recently as follows -  
(Shells numbered smaller  $\rightarrow$  larger diameter).

#1	8/29	Exp 259
#2+1	8/26	258
#1,2,3,4	8/25	257
#1 $\rightarrow$ 6mc	8/24	256
#1 $\rightarrow$ 16mc	8/24	255

In all cases sample tank filled to top with H<sub>2</sub>O. Leak would be into reactor.

Zeuth  
rdg

#26 A	no wipe test	concrete side	area wiped = 1/2 ft <sup>2</sup>
#9 B	"	"	"
#5 B	6000 $\alpha$ /m wipe	"	"
#1 B	600	"	"
#1 A	> 1000 $\alpha$ /m	"	"
#7 B	no wipe test	"	"
#6 B	"	"	"
#5 B	~ 100 $\alpha$ /m	concrete side	"

(#24 was verified that contamination is  $\alpha$ )

These indicate #1-4 were used during a leak - concrete side #5 contaminated (from #4).

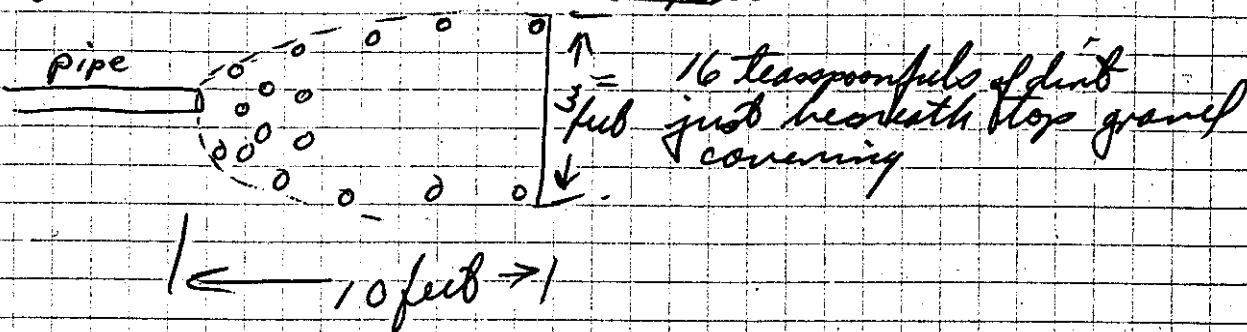
Spot chemical test of water from al shells,  $\rightarrow$  U neg.

Removed top sample - noted blisters in Al lith on bottom of reactor & black corrosion where lateral (vertical) surface is welded to bottom.

64 9/6/49

Samples of dirt taken from end of drain line

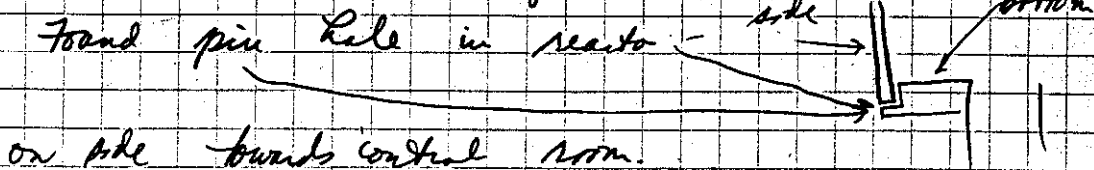
o - - - - - fence



Samples taken in tared beaker (377 gm) and placed in oven to get bone dry wgt.

Sample consists of limestone chip, organic matter, clay and cinder. A count over this area 500 -> 10000 <math>\times</math> / mi - (See pg 66 for counts)

Removed Al shells - found heavy encrustation of yellow uranium salt - under them some black on deposit surfaces in contact with Al. Pallid paint blistered on exterior & yellow surfaces of reactor.



Discharge end of 3" drain line - showed zinc wipe test on upper surface (inside) high count ( $\sim 10^4$ ) on bottom inside surface

It seems unlikely that the acid deposit under the Al H<sub>2</sub>O shells could have been formed in the presence of water - unless a precipitate occurred upon reaction of UO<sub>2</sub>(NO<sub>3</sub>)<sub>2</sub> with water. (The precipitate was rather less soluble in Na<sub>2</sub>CO<sub>3</sub> than expected).

The bottom (inside) of the 8" reactor

9/6/49

65

is distal away from center:



due to poor manufacture & provides a  
hold up. <sup>(2,100 cc)</sup> for solution. It may

be that

upon examination the bottom was dry  
& not encrusted - It may be that

the large block developed at end of

Exp 261 & then ~~held up~~

upward during the period of  
inactivity 9/30 → 9/6.

9/7 -

Spgr A = 1.549  
K = 1.550

sampled storage cylinders A & K in order to  
determine if significant amounts of water had  
leaked into solution during those times  
when sump water level was above solution  
level (normalized for density differences).

Removed & cleaned all mugs used to  
support s. steel & all shells -

Removed 5' of 3" pipe & 45° ell from  
end of discharge (H.W.) line in order to fit pipe  
cap having 3/8" flange connection to 3"  
line. With s. steel centrifugal motor &  
piston valve circulated hot concentrated  $\text{Na}_2\text{CO}_3$   
through 3" line. Removed & replaced with  
fresh  $\text{CO}_2$  solution which stood in pip over  
night.

Took each sample to lab.

Benny & Giffner here - they are to determine  
shells and other parts we can take to 1410 &  
maintain material balance.

DC

8/8 - Washed all shells -  $\text{HNO}_3$  - to  $\sim 3000$  residual  $\alpha$ . Took # 1410  
 checked S. Shell shells with Poppy - maximum count found  $\sim 1-2000$ .  
 Stencilled numbers on a.o. shells & took 1-6 B ac, 1-7 A ac to 1410  
 checked rest of a.o. shells - no contamination.  
 Completed working of 3' pipe - 2' of carbonate wash -  
 followed by  $\text{HNO}_3$  - 1:1 - followed by water.  
 Decontaminated inside of temp tank -  
 Sampled solution for decontamination.  
 Cleaned out 3' dump sector - workshop for water - removed water -

9/9 - all SS back from 1410 - S.S. not clean -  
 300 wraps -

Sampled ground at 3' discharge - trace shown  
 $\sim 10^4$   $\alpha/\text{mi}$  - with high pressure argon chamber -  $0.15 \text{ gm}/\text{ft}^2$  -

Surface -  $5000$   $\alpha/\text{mi}$ . No response on 8 chamber.  
 (Drip line beside K1095 - river end  $\sim 5000$   $\alpha/\text{mi}$ )

3' pipe showed  $\sim 8$  on high pressure argon chamber - ( $0.29 \text{ gm}/\text{ft}^2$ )

It

[Surface of limestone, drip line of gutter: 11x1 on 2 ends -  
 $\frac{1}{2}$ " below surface 3x1 on 2 ends]

1" below surface  $\frac{5}{8}$ " from discharge of 3' line - no  $\alpha$

Indications are that the limestone strongly  
 absorbed the U.

8' sector to 1410 -

Removed gravel from  $\sim 75 \text{ ft}^2$  at discharge of  
 3' line - total of about  $10 \text{ ft}^3 \approx 10^3 \text{ lbs}$ .

Results from analysis of gravel (see pg 64):  $\frac{0.9}{0.09} \mu\text{g}/\text{gm}$  rocks  
 On this basis the  $10 \text{ ft}^3$  removed today contains  $\sim 0.05 \text{ gm}$ .  $0.5 \text{ gm}$ .



9/12 - men from 1410 helped decontaminate spools & ~~also~~  
at shells -

9/13 Decontaminated & replaced 5' length of 3' pipe.

9/14 Profess & Menz at FOS; reviewed spill; they  
suggested obtaining analyses of samples taken from  
ground —

SECRET

A }  
23 }  
3 }

D }  
24 }  
4 }

SECRET

18 }  
9 }  
7 }

---

20 } B  
1 }  
Zack

SECRET

SECRET