



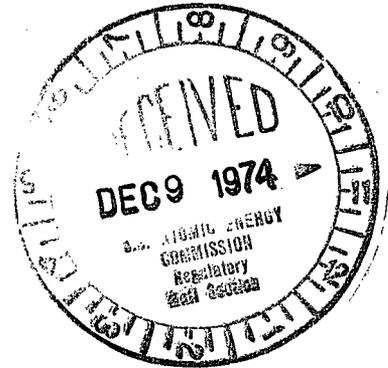
UNION CARBIDE CORPORATION

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STERLING FOREST
RESEARCH CENTER

December 5, 1974



U. S. Atomic Energy Commission
Materials and Plant Protection Branch
Directorate of Licensing
Washington, D. C. 20545

Attn: Mr. R. G. Page, Chief

Ref: (a) USAEC letter L:MPP:CNS 70-687 of 11/12/74

Dear Mr. Page:

The supplemental information for the Union Carbide Material Control and Accounting Plan which was requested in your letter referenced above is enclosed.

All of the information has been provided as requested except for the provisions in the measurement QC program requiring weekly standard measurements and fifteen replicate process measurements for each type of measurement carried on in our process. We respectfully submit that the proposed QC program herewith submitted will be sufficient to preclude the development of a significant MUF. The quantity of material which is handled in the operation at the Union Carbide Corporation Sterling Forest Laboratory is relatively small and the proposed limits of error in measurement would not result in a discrepancy involving a large quantity of SNM.

Very truly yours,

James J. McGovern
Manager
Radiochemical Production



JJMcG:js
Enclosures

2361

SUPPLEMENTAL INFORMATION
UNION CARBIDE MATERIAL CONTROL AND ACCOUNTING

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SUPPLEMENTAL INFORMATION ON MATERIAL CONTROL & ACCOUNTING PLAN

GENERAL:

The below information is to be incorporated in the Union Carbide Corporation Material Control and Accounting Plan. This supplemental information should be incorporated in the plan according to the topic numbers listed.

2.2 Samples of a Master Log sheet and a Material Balance Area Log sheet are enclosed as Appendix "E". When material is received on site, it is recorded in the Master Log and the MBA Log for the UO₂ Feed MBA. Entries are identified by date and shipper's batch number. When material is transferred internally between MBA's, concurrent entries are made in the MBA Log of the areas from and to which the material is transferred as well as the Master Log. The SNM being transferred between MBA's is identified as follows:

<u>Source MBA</u>	<u>Receiving MBA</u>	<u>Method of Identity</u>
UO ₂ Feed	Plating, Encapsulating Quality assurance.	Supplier's batch No. or serial No.
Plating Encapsulation Quality assurance	Reactor Operations	Target number
Reactor Operations	Radiochemical Operations	Target number

2.4 The AEC Form 741 containing the necessary analysis data for measurements will be retained by the SAO. These records will be retained for a period of at least 5 years.

5.5 In preparation for each inventory a meeting will be convened to list materials on inventory by code number according to material form and batch number.

For example:

- UO₂ Feed MBA - F (1 → n)
- P, E, & QC MBA - P (1 → 4) for plating batches
- Pw (1 → n) for plating waste
- T (1 → n) for sealed targets
- Reactor Operation - T (1 → n) for sealed targets
- Radiochemical Processing - Rw (1 → n)

The material custodians from each MBA will be present at the meeting to insure all materials are included in the inventory list.

5.11.1 a) The total uranium content for Items 1 and 2 (uranyl nitrate plating solutions and waste solutions from the feed and plating areas) will definitely be measured using the Chemical Analytical method described in Appendix F. The solutions will be collected into separate bottles. The volumes of the feed and stock solutions will be measured by graduate cylinders and then placed into bottles labeled with appropriate codes and indicating original volume. The volume of the waste solutions from feed and plating solution will be measured by graduate cylinders. These solutions will also be placed in bottles labeled with appropriate codes and volume content. Quality Control will remove bulk taps from these solutions to use in their measurement of total uranium and ^{235}U .

The final total uranium content for Item 4 (waste solutions removed from the hot cells) will be attempted using this same technique but on a small aliquot of the bulk due to the radiation hazard involved. Item 4 has not yet been attempted and will require a pre-radiochemical separation of the uranium from other fission waste. This pre-separation of uranium will be accomplished using the procedure described in Appendix G. The waste from several waste batches will be combined in liquid form, mixed in a hot cell, and its volume will then be measured in a precalibrated volumetric vessel inside a hot cell. A sample of this will be removed for analysis and the solution will be immediately solidified and placed in a waste drum. The sample will be delivered to Quality Control and an aliquot of this bulk tap (~ 0.1 ml), will be used for the uranium determination.

The total uranium content of Item 3 (Targets) cannot be measured because the targets are sealed for irradiation. The ^{235}U content, however, will be measured using nondestructive techniques described in 5.11.1 d).

5.11.1 b) The Quality Control department will attempt to measure the ^{235}U content of the hot cell waste using the technique described in 5.11.1 c). An aliquot of the waste solution will be chemically separated to remove the uranium from other fission products, as described in Appendix G. The separated solution will then be counted for ^{235}U in the same manner as other solutions.

5.11.1 c) The nondestructive radiometric assay for ^{235}U will be completed using a 3" x 3" NaI detector connected to a multichannel analyzer and its associated software. Aliquots of the solutions to be assayed will be counted on the base (~ 0.635 cm geometry-to-crystal base) of the 3" x 3" geometry stand for 5 minutes live time on a channel range of 2.5 Kev/ch over 400 channels. The photopeak area of the 185 Kev gamma ray will be computed (using the method of Covallis) and compared, after correcting for the aliquot taken, to standard data. All volumes to be counted will be adjusted to a final volume of 3.0 ml in our standard lucite disposable vials. The volume of 3 ml allows us to use small taps from 20 λ for concentrated solutions to a full 3 ml for dilute solutions.

Standards for these measurements were procured from NBS in the form of U_3O_8 . They are NBS-U930, U900, and U850. This material was given to our site Analytical Services to make solutions, of 1:5 HNO₃, having ~ 1 gm of each standard per 100 ml. Figure 1 shows analytical report of this preparation. Aliquots of these solutions were taken and a standard curve prepared from the data in Table I showing gms ^{235}U vs. photopeak area (C/5m) Figure 2. (Normalized values from this data is shown in Table IA.) At the same time a known aliquote from the U930 solution was taken and flame-sealed in an NBS flame-seal vial. This flame-sealed vial will be used as a sealed control sample. the count rate of this vial was recorded (Table II) and will be used to determine analyzer calibration and detector efficiency and/or drift each time liquid measurements are made. A control chart will be developed and each subsequent count made with this standard will be recorded to determine any long-term variations.

At the same time a NBS ^{137}Cs point source standard was counted at 10 cm geometry, this is our standard source used for all other radiometric measurements, and it will be used as a secondary check of the system using the control chart method. (See Appendix H)

5.11.1 d) Four standard targets were taken at random from the target inventory. The targets were nondestructively counted for their ^{235}U content using a geometry of 32 cm (fixed by geometry stand on 3" x 3" crystal) and a count time of 5 minutes live time. Table III shows results of this counting. Three of the targets - No. 706, No. 503, and No. 333 - were then stripped of their total uranium by cutting

LAB. NO. 11-112
DATE REC'D 11/15/74

MATERIAL- Prepave 3 Uranium
Salts
SUBMITTED BY- H. W. Nass U-850
SOURCE-

ANALYZE FOR	APPROX. % EXPECTED	% REPORTED	ANALYST	BOOK
		1.1557g/100ml	UT	928/98
		1.1522g/100ml		
		1.1714g/100ml		

ACCOUNT NO.- 537
REPORT TO- H. W. Nass U-930
BUILDING NO.- 4
DATE- 11/15/74

REMARKS- Weigh accurately,
dissolve ^{HNO3} and dilute to 100
ml (1-5 HNO3)

CHECKED BY: RS.

REPORTED BY: UT

DATE: Nov. 19, 1974

FIGURE 1

TABLE I

PREPARATION OF STANDARD LIQUID CURVE DATA*

0.009248 g/ml of 93.276 wt% ²³⁵U
500λ tap (0.004624 g) c/5 min

Count No.	No. 1	No. 2	No. 3	\bar{X}	$\sigma \pm \%$
1	13799	13661	14093	13851	220
2	11960	12688	13964	12871	1014
3	14072	14219	13307	13866	490
4	13542	13330	13179	13350	182
5	13434	14054	13389	13626	372
\bar{X}	13712	13590	13586	13513	131
σ	285 \pm 2.1%	612 \pm 4.5%	413 \pm 3.1%		

Total $\bar{X} = 13623$ $\sigma = 436$, $2\sigma = 872$ or $\pm 6.4\%$
 200λ tap (0.001850 g)

	No. 1	No. 2	No. 3	\bar{X}	σ
1	5862	5979	5739	5860	120
2	5965	5957	5756	5893	118
3	6012	5558	6025	5865	266
4	5751	5801	6046	5866	158
5	5602	5869	5702	5724	135
\bar{X}	5838	5833	5854		
σ	166	169	167		

Total $\bar{X} = 5842$ $\sigma = 155$ $2\sigma = 310$ or $\pm 5.3\%$
 20λ tap (0.0001850 g)

	No. 1	No. 2	No. 3	\bar{X}	σ
1	643	670	527	613	76.0
2	531	639	495	555	74.9
3	592	582	486	553	58.5
4	556	632	407	594	54.0
5	749	526	548	537	16.0
\bar{X}	580	609	514		
σ	48.6	56.5	28.7		

Total $\bar{X} = 568$ $\sigma = 72.5$ $2\sigma = 144.9$ or $\pm 25.9\%$

*All data taken in 3 ml of solutions on Base with 3" x 3" NaI crystal.

TABLE I

PREPARATION OF STANDARD LIQUID CURVE DATA0.008787 g/ml of 90.098 wt% ²³⁵U200λ tap (0.001757 g) c/5 min

Count No.	No. 1	No. 2	No. 3	\bar{X}	σ
1	5454	5056	5386	5299	212
2	5206	5232	5362	5267	83.6
3	5473	5378	5168	5340	156
4	5212	5255	5695	5387	267
5	4927	4913	4884	4908	21.9
\bar{X}	5254	5167	5299	5240	87.1
σ	223	183	299		

Total \bar{X} = 5240 σ = 229 2σ = 458 or $\pm 8.7\%$

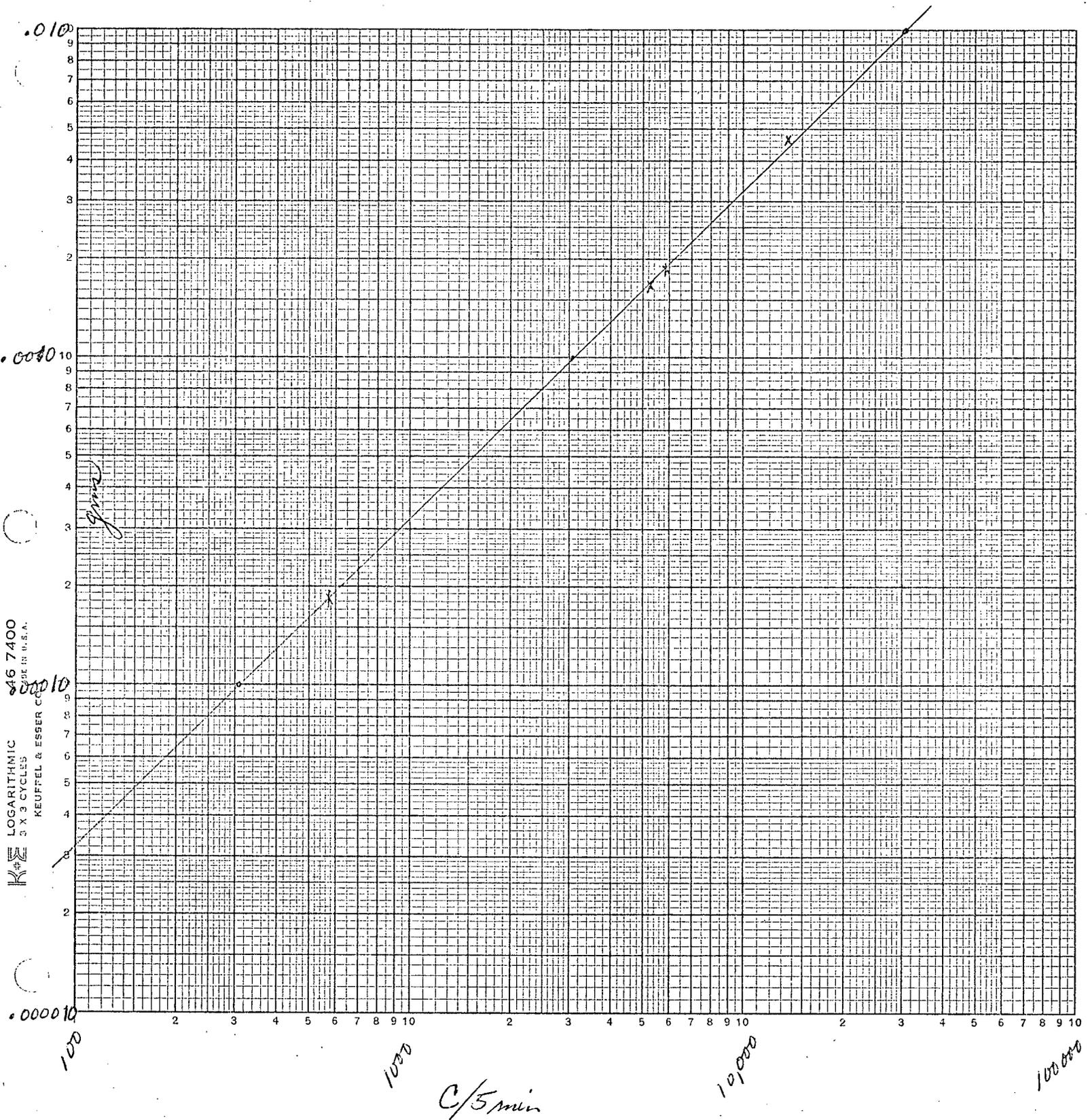
0.008315 g/ml of 84.988 wt% ²³⁵U200λ tap (0.001663 g) c/5 min

Count No.	No. 1	No. 2	No. 3*	\bar{X}	σ
1	5074	5154	4712		
2	5419	5430	4329		
3	5210	5096	4906		
4	5305	5300	5095		
5	5290	5470	4765		
\bar{X}	5260	5290	4761		
σ	128	164	284		

Total \bar{X} = 5275 σ = 140 2σ = 280 or $\pm 5.3\%$

*This column average is 9.7% below the average of the other two samples and was considered in error and not used.

Figure II
 Plot gms ^{235}U Vs Counts/5 minute
 Calibration for liquid Uranium Sources



LOGARITHMIC
 5 X 3 CYCLES
 KEUFFEL & ESSER CO.
 MADE IN U.S.A.
 46 7400

Note: x are from actual values
 o are from average value calculation

TABLE IA

Normalized Counts/5 min/g ^{235}U

<u>^{235}U Source</u>	<u>g ^{235}U</u>	<u>c/5 m</u>	<u>c/5 m/g</u>
93.276%	.004624	13623	2.9461×10^6
"	.001850	5842	3.1585×10^6
"	.0001850	568	3.0709×10^6
90.098%	.001757	5240	2.9817×10^6
84.988%	.001663	5275	3.1720×10^6

$$\bar{X} = 3.0658 \times 10^6$$

$$\sigma = 0.1016 \times 10^6$$

$$\text{or } \pm 3.3\%$$

$$2\sigma = 0.17225 \times 10^6$$

$$\text{or } 6.64\%$$

TABLE IISEALED CONTROL AND CALIBRATION STANDARDSNBS ^{137}Cs - $^{137\text{m}}\text{Ba}$ Sealed Point Source No. 82

163008

162676 $\bar{X} = 163113$ 163307 $\sigma = 330$ or 0.2%163028 $2\sigma = 660$ or 0.4%

163546

Prepared Uranium Standard Solution in Flame Sealed Vial500 λ of 93.276 wt% ^{235}U , 0.0109264 g/ml

	<u>Vial 1</u>	<u>Vial 2</u>
	11676	11838
	11575	
	11786	11284
	11642	11838
	<u>11251</u>	<u>11786</u>
$\bar{X} =$	11586	$\bar{X} = 11686$
$\sigma =$	202 or $\pm 1.8\%$	$\sigma = 270 \pm 2.3\%$
$2\sigma =$	404 or $\pm 3.5\%$	$2\sigma = 540 \pm 4.6\%$

TABULATION OF S.N.M. TARGET TUBE

COUNT RATE DATA FOR 185 Kev GAMMA OF ^{235}U

		<u>Target Capsule 706</u>					
		<u>A^(a)</u>	<u>B</u>				
(b)	1	162047	152957	$\bar{X} = 158193$			
	2	150682	164524	$\sigma = 5971$ or $\pm 3.77\%$			
	3	159138	163269	$2\sigma = 11942$ or $\pm 7.55\%$			
	4	162713	150211				
<u>Target Capsule 503</u>							
		<u>A</u>	<u>B</u>				
	1	78047	74748	$\bar{X} = 74914$			
	2	75419	72161	$\sigma = 2304$ or $\pm 3.08\%$			
	3	71766	75503	$2\sigma = 4608$ or $\pm 6.15\%$			
	4	77774	73895				
<u>Target Capsule 333</u>							
<u>POSITION A</u>				<u>POSITION B</u>			
<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
107240	104657	99966	101371	104618	102543	102799	101747
100731	109062	107770	103196	103526	108825	107992	104270
102957	105402	106040	105014	105791	110774	104514	102932
105738	102612	104639	102762	104678	107006	101402	98850
\bar{X}	\bar{X}	\bar{X}	\bar{X}	\bar{X}	\bar{X}	\bar{X}	\bar{X}
104167	105433	104604	103086	104653	107287	104177	101950
σ	σ	σ	σ	σ	σ	σ	σ
2897	2691	3346	1503	925	3517	2844	2309
Overall $\bar{X} = 104419$				$\sigma = 2762.6$ or $\pm 2.65\%$			
				$2\sigma = 5525.0$ or $\pm 5.29\%$			
<u>Target Capsule 804</u>							
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>\bar{X}</u>	<u>σ</u>	<u>2σ</u>
Position A	164024	165929	169198	163393	165636	2608	5216
Position B	170722	162986	164036	171854	167399	4534	9068
Overall $\bar{X} = 166518$					$\sigma = 3552$ or $\pm 2.13\%$		
					$2\sigma = 7104$ or $\pm 4.27\%$		

NOTE: (a) Position A, nipple of target tube facing left; Position B, nipple facing right.
 (b) 1, 2, 3 and 4 refer to 1/4 turns in axial rotation.

open and having the Analytical laboratory chemically dissolve the uranium. Each target was stripped twice with each stripping going into separate flasks and adjusted to a known volume. Taps of these solutions were then compared to the liquid standards as in 5.11.1 c). By counting in duplicate the total gms of ^{235}U for each target were determined and compared to the counts/5 minutes of the integrated photopeak area for the 185 Kev γ of ^{235}U obtained by the whole target counting, thus establishing a calibration curve for the ^{235}U content of the sealed target tubes. The target solution data are shown in Table IV, the determined total gms in Table V, the counts/5 minutes/gm ^{235}U in the tube in Table VI. The standard curve from this data is shown in Figure 3.

The fourth tube, No. 804, was counted at the same time but it was not destroyed and it will be used as a control standard for both calibration and control purposes. The integrated PPA c/5m will be recorded and plotted on a control chart each time it is counted to establish any drifts or changes in detector and analyzer systems (Figure 4). (See Appendix J)

5.11.2 The random and systematic error uncertainty for each measurement is shown in Appendix D.

6.1 A corrected materials diagram is enclosed as Appendix C.

6.2 Measurements - a corrected 6.2 is submitted to add the analysis of U as well as U^{235} in the radioactive waste material solidified for shipment.

6.2.1 Uranium analysis for materials in solution will be done in accordance with the procedure described in 5.11.1 a).

6.2.2 U^{235} analysis for materials in solution will be done in accordance with the procedure outlined in 5.11.1 b) and c).

6.2.3 Uranium analysis for material in sealed targets cannot be done, however prior measurements from plating solutions will be used for element content of targets.

6.2.4 U^{235} analysis of sealed targets will be done in accordance with the procedure described in 5.11.1 d).

6.3 A tabulation of errors is shown in Appendix D.

6.4 A demonstration of LEMUF is shown in Appendix D.

TABLE IV

COUNT RATE DATA FROM ^{235}U SOLUTIONS STRIPPED FROM TARGETS (c/5 min)Tube No. 333, 1 ml Taps from 1000 ml volume

<u>Count No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
1	18968	19947	18984	18957	17548
2	18649	17626	17402	18939	19803
3	19007	18576	19043	19025	17493
\bar{X}	18874	18716	18476	18973	18281
Overall	$\bar{X} = 18664$		$\sigma = 801$	$2\sigma = 1602$ or $\pm 8.6\%$	

Tube No. 503, 1 ml Taps from 1000 ml Volume

<u>Count No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
1	13520		13053	12290	12580
2	13140		12992	13647	12529
3	<u>12678</u>		<u>12938</u>	<u>13096</u>	<u>12637</u>
\bar{X}	13111		12994	13013	12582
Overall	$\bar{X} = 12925$		$\sigma = 404$	$2\sigma = 808$ or 6.3%	

Tube No. 706, 1 ml Taps from 1000 ml volume

<u>Count No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
1	27763	26559	29233	24451	25261
2	28528	26996	29871	28598	28239
3	30148	28756	30916	30256	26146
\bar{X}	28813	27437	30006	27768	26548
Overall	$\bar{X} = 28114$		$\sigma = 1900$	$2\sigma = 3800$ or $\pm 13.5\%$	

TABLE VDETERMINATION OF ^{235}U IN TARGET TUBES

<u>Target Tubes</u>	<u>c/ml/5m</u>	<u>Total Vol.</u>	<u>c/5m/g ^{235}U</u>	<u>Total gms ^{235}U</u>
333	18664	1000 ml	3.0658×10^6	6.0878
503	12925	1000 ml	"	4.2159
706	28114	1000 ml	"	9.1702

$$\frac{\text{c/ml/5m} \times 1000 \text{ ml}}{\text{c/5m/g } ^{235}\text{U}} = \text{gms } ^{235}\text{U}$$

TABLE VIDETERMINATION OF c/5m/g. ^{235}U FOR TARGET TUBESCOUNTED AT 32 Cm ABOVE 3" x 3" NaI DETECTOR

<u>Target Tube</u>	<u>C/5m/Tube</u>	<u>gms ^{235}U</u>	<u>C/5m/g. ^{235}U</u>
333	104419	6.0878	1.7152×10^4
503	74914	4.2159	1.7769×10^4
706	158193	9.1702	1.7251×10^4

$$\bar{X} = 1.7391 \times 10^4$$

$$\sigma = 0.03314 \times 10^4 \text{ or } \pm 1.9\%$$

Figure III

Control Chart for ²³³U Fume Sealed Vial # 1
Counts/5 minutes on Beta - 3 x 3 NaT

GRAPH PAPER GRAPHIC CONTROLS CORPORATION Buffalo, New York
Printed in U.S.A.
SQUARE 10 X 10 TO THE CENTIMETER AS 8014-01

12000
11900
11800
11700
11600
11500
11400
11300
11200
11100
11000

11/20/54

+3%

Aug

-3%

Control Graph for Target = 804

$\bar{x} = 166516$
C/min

+5% = 174844
-5% = 158188

Control limits $\pm 2.3\sigma$

GRAPHIC CORPORATION Buffalo, New York
Printed in U.S.A.
SQUARE 10 X 10 TO THE CENTIMETER AS-8014-GF

180000
178
176
174
172
170000
168
166
164
162
160000
158
156
154
152
150000

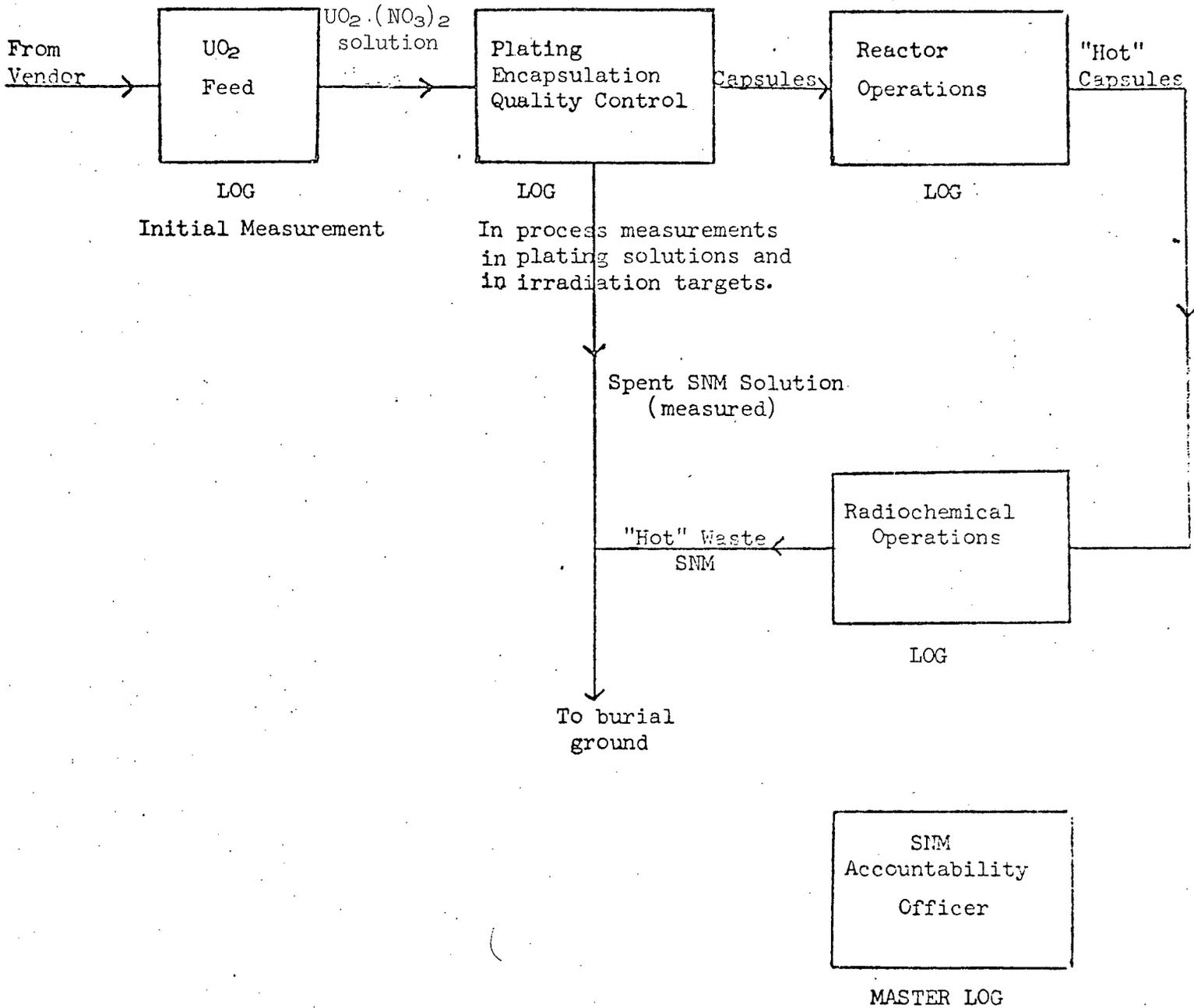
+5%

\bar{x}

-5%

1/2/74

6.5 The measurement quality control program will consist of checking both the uranium and ^{235}U measurement techniques against known NBS standards at least once during each material balance period. The analysis results will be recorded for determination of any long term bias. Bias corrections, if required, will be made annually. The inventory turnover is so rapid in this plant process that bias corrections will only be made to inventory on hand which had been previously measured.



PLANT PROCESS FLOW DIAGRAM

(Each block in flow chart represents a material balance area.)

APPENDIX D

LEMUF MODEL

TYPICAL CHART OF MATERIALS FOR LEMUF DETERMINATION

Type of Material and Location	MUF Component	Typical Quantity		Item Identity
		U	²³⁵ U	
Material from Uranyl Nitrate Feed	BI	376.3	350 gms	(BI _{f1})
		376.3	350 gms	(BI _{f2})
	R	376.3	350 gms	(R _{f3})
		376.3	350 gms	(R _{f4})
	EI	376.3	350 gms	(EI _{f4})
Uranyl Nitrate Plating	BI	53.8	50 gms	(BI _{p1})
		53.8	50 gms	(BI _{p2})
		53.8	50 gms	(BI _{p3})
		53.8	50 gms	(BI _{p4})
	EI	48.4	45 gms	(EI _{p5})
		48.4	45 gms	(EI _{p6})
		48.4	45 gms	(EI _{p7})
		48.4	45 gms	(EI _{p8})
Uranyl Nitrate Plating Waste	BI	10.75	10 gms	(BI _{pw1})
	S	75.3	70 gms	(S _{pw1})
	EI	10.75	10 gms	(EI _{pw1})
UO ₂ Targets	BI	430.1	400 gms	(BI _{t1} → 40)
	EI	322.6	300 gms	(EI _{t50} → 80)
Uranyl Sulfate Radioactive Waste	BI	1032.3	960 gms	(BI _{rw1-48})
	S	376.3	350 gms	(S _{rw1})
		376.3	350 gms	(S _{rw2})
	EI	1032.3	960 gms	(EI _{rw40-88})

APPENDIX D, CONT'D.

MEASUREMENT ERROR STANDARD DEVIATIONS

<u>Location and Type of Material</u>	<u>Relative Systematic Standard Dev.</u>	<u>Relative Random Standard Dev.</u>
<u>Volume Determination Element & Isotope</u>		
	<u>$\sigma_{\delta i}$</u>	<u>$\sigma_{\epsilon i}$</u>
UO ₂ feed (Uranyl Nitrate Solution)	.001	.005
Plating solution (Uranyl Nitrate)	.02	.03
Plating waste batch (Uranyl Nitrate)	.001	.006
Radioactive waste batch (Uranyl Sulfate) shipped	.02	.03
Radioactive waste solution in cell (targets)	0	0
Targets (UO ₂)	0	0
<u>Element Sampling</u>		
	<u>$\sigma_{\Delta i}$</u>	<u>$\sigma_{\eta i}$</u>
UO ₂ feed solution	.02	.01
Plating solution	.006	.01
Plating waste solution	.006	.01
Radioactive waste solution shipped	.006	.07
RW solution in cell (targets)	.006*	.01*
Targets	.006*	.01*
<u>Element Analysis</u>		
	<u>$\sigma_{\epsilon k}$</u>	<u>$\sigma_{\omega k}$</u>
UO ₂ feed solution	.004	.01
Plating solution	.004	.01
Plating waste solution	.004	.01
Radioactive waste solution shipped	.004	.07
RW solution in cell (targets)	.004*	.01*
Targets	.004*	.01*
<u>Isotope Sampling</u>		
	<u>$\sigma_{\lambda 1}$</u>	<u>$\sigma_{\mu 1}$</u>
UO ₂ feed solution	.02	.01
Plating solution	.02	.01
Plating waste solution	.02	.01
Radioactive waste solution shipped	.02	.07
Radioactive waste solution in cell (targets)	0*	0*
Targets	0	0

*Previous measurements from plating solutions or targets.

APPENDIX D, CONT'D.

MEASUREMENT ERROR STANDARD DEVIATIONS

<u>Location and Type of Material</u>	<u>Relative Systematic Standard Dev.</u>	<u>Relative Random Standard Dev.</u>
<u>Isotope Analysis</u>	<u>σ_y</u>	<u>σ_r</u>
Materials in solution (F, P, PW, RW shipped)	.009	.045
Radioactive waste solution in cell (targets)	.04*	.04*
Targets	.04	.04

*Previous measurements from plating solutions or targets.

APPENDIX D, CONT'D.

Product
C
σ² (gm²)

Variance in Volume Measurements for Element Determinations

$$C_{\delta_1} = (BI_{f_1} + BI_{f_2} + R_{f_3} + R_{f_4} - EI_4 + BI_{pw_1} - S_{pw_1} - EI_{pw_1})^2 \times (.001)^2 =$$

$$C_{\delta_2} = (BI_{p_1 \rightarrow 4} - EI_{p_5 \rightarrow 8} - S_{rw_1-2})^2 \times (.02)^2 =$$

$$C_{\epsilon_1} = (BI_{f_1})^2 + (BI_{f_2})^2 + (R_{f_3})^2 + (R_{f_4})^2 + (-EI_{f_4})^2 \times (.005)^2 =$$

$$C_{\epsilon_2} = (BI_{p_1})^2 + \dots + (BI_{p_4})^2 + (-EI_{p_5})^2 + \dots + (-EI_{p_8})^2 + (-S_{rw_1})^2 + (-S_{rw_2})^2 \times (.03)^2 =$$

$$C_{\epsilon_3} = (BI_{pw_1})^2 + (-S_{pw_2})^2 + (-EI_{pw_1})^2 \times (.006)^2 =$$

Variance in Sampling for Element Determinations

$$C_{\Delta_1} = (BI_{f_1+2} + R_{f_3+4} - EI_{f_4})^2 \times (.02)^2 =$$

$$C_{\Delta_2} = (BI_{(p_1, 2, 3+4)} - EI_{(p_5, 6, 7+8)} + BI_{pw_1} - S_{pw_1} - EI_{pw_2} - S_{rw_1+2} + BI_{t_1 \rightarrow 40} - EI_{t_{50} \rightarrow 80} + BI_{rw_1-48} - EI_{rw_{40-88}})^2 \times (.006)^2 =$$

$$C_{\eta_1} = (BI_{f_1+2} + R_{f_3+4} - EI_{f_4} + BI_{p_1, 2, 3+4} - EI_{p_5, 6, 7+8} + BI_{pw_1} - S_{pw_1} - EI_{pw_1} + BI_{t_1 \rightarrow 40} - EI_{t_{50} \rightarrow 80} + BI_{rw_1-48} - EI_{rw_{40-88}})^2 \div \text{No. Samples} \times (.01)^2 =$$

$$C_{\eta_2} = (-S_{rw_2+2})^2 \div \text{No. Samples} \times (.07)^2 =$$

APPENDIX D, CONT'D.

Variance in Element Analysis Determination

Product
C_o² (gm²)

$$C_{\theta_1} = (BI_{f_{1+2}} + R_{f_{3+4}} - EI_{f_4} + BI_{p_{1,2,3+4}} - EI_{p_{5,6,7+8}} + BI_{p_{w1}} - S_{p_{w1}} - EI_{p_{w1}} + BI_{t_{1 \rightarrow 40}} - EI_{t_{50 \rightarrow 80}} + BI_{rw_{1 \rightarrow 48}} - S_{rw_1} - S_{rw_2} - EI_{rw_{40 \rightarrow 88}})^2 \times (.004)^2 =$$

$$C_{\omega_1} = (BI_{f_{1+2}} + R_{f_{3+4}} - EI_{f_4} + BI_{p_{1 \rightarrow 4}} - EI_{p_{5 \rightarrow 8}} + BI_{p_{w1}} - S_{p_{w1}} - EI_{p_{w1}} + BI_{t_{1 \rightarrow 40}} - EI_{t_{50 \rightarrow 80}} + BI_{rw_{1 \rightarrow 48}} - EI_{rw_{40 \rightarrow 88}})^2 \div \text{No. Analyses} \times (.01)^2 =$$

$$C_{\omega_2} = (-S_{rw_{1+2}})^2 \div \text{No. Analyses} \times (.07)^2 =$$

APPENDIX D (Cont'd)

Product
C_σ² (gm²)

Variance in Volume Measurements for Isotope Determinations

$$\begin{aligned}
 C_{\delta_1} &= \text{Same as } C_{\delta_1} \text{ for element determinations only} \\
 &\quad \text{isotope weights substituted} \quad \times (.001)^2 \quad = \\
 C_{\delta_2} &= \text{Same as } C_{\delta_2} \text{ for element determinations only} \\
 &\quad \text{isotope weights substituted} \quad \times (.02)^2 \quad = \\
 C_{\epsilon_1} &= \text{Same as } C_{\epsilon_1} \text{ for element determinations only} \\
 &\quad \text{isotope weights substituted} \quad \times (.005)^2 \quad = \\
 C_{\epsilon_2} &= \text{Same as } C_{\epsilon_2} \text{ for element determinations only} \\
 &\quad \text{isotope weights substituted} \quad \times (.03)^2 \quad = \\
 C_{\epsilon_3} &= \text{Same as } C_{\epsilon_3} \text{ for element determinations only} \\
 &\quad \text{isotope weights substituted} \quad \times (.006)^2 \quad =
 \end{aligned}$$

Variance in Sampling for Isotope Determination

$$\begin{aligned}
 C_{\lambda_1} &= (BI_{f_{1+2}} + R_{f_{3+4}} - EI_{f_4} + BI_{p_{1 \rightarrow 4}} - EI_{p_{5 \rightarrow 8}} \\
 &\quad + BI_{pw_1} - S_{pw_1} - EI_{pw_1} - S_{rw_{1+2}})^2 \times (.02)^2 \quad = \\
 C_{\mu_1} &= (BI_{f_{1+2}} + R_{f_{3+4}} - EI_{f_4} + BI_{p_{1 \rightarrow 4}} - EI_{p_{5 \rightarrow 8}} \\
 &\quad + BI_{pw_1} - S_{pw_1} - EI_{pw_1})^2 \div \text{No Samples} \times (.01)^2 \quad = \\
 C_{\mu_2} &= (S_{rw_{1+2}})^2 \div \text{No Samples} \times (.07)^2 \quad =
 \end{aligned}$$

Variance in Analysis for Isotope Determination

$$\begin{aligned}
 C_{\gamma_1} &= (BI_{f_{1+2}} + R_{f_{3+4}} - EI_{f_4} + BI_{p_{1 \rightarrow 4}} - EI_{p_{5 \rightarrow 8}} \\
 &\quad + BI_{pw_1} - S_{pw_1} - EI_{pw_1} - S_{rw_{1+2}})^2 \times (.009)^2 \quad = \\
 C_{\gamma_2} &= (BI_{t_{1 \rightarrow 40}} - EI_{t_{50 \rightarrow 80}} + BI_{rw_{1-48}} - EI_{rw_{40 \rightarrow 88}})^2 \\
 &\quad \times (.04)^2 \quad =
 \end{aligned}$$

APPENDIX D (Cont'd)

Product
 C_{σ^2} (gm²)

$$C_{V_1} = (BI_{f_{1+2}} + R_{f_{3+4}} - EI_{f_4} + BI_{P_{1 \rightarrow 4}} - EI_{P_{5 \rightarrow 8}} + BI_{pw_1} - S_{pw_1} - EI_{pw_1} - S_{rw_{1+2}})^2 \div \text{No Samples} \times (.045)^2 =$$

$$C_{V_2} = (BI_{t_{1-40}} - EI_{t_{50 \rightarrow 80}} + BI_{rw_{1-48}} - BI_{rw_{40 \rightarrow 88}})^2 \div \text{No. Samples} \times (.04)^2 =$$

SUMMARY of Variance Components for Element Determination:

<u>Operation</u>	<u>Error Type</u>	<u>Variance</u>
Volume Measurement	LT Systematic + random	C_{σ^2}
Sampling	LT Systematic + random	"
Analysis	LT Systematic + random	"
	Total Variance	(gm^2)
	LEMUF = 2(total variance) ^{1/2}	

SUMMARY of Variance Components for Isotope Determination:

Volume	Systematic + random	C_{σ^2}
Sampling	Systematic + random	"
Analysis	Systematic + random	"
	LEMUF =	2(total variance) ^{1/2}

LEMUF DETERMINATION WITH SUBSTITUTED TYPICAL VALUESVariance in volume measurements for elementC σ^2

$C_{\delta_1} = (376.3 + 376.3 + 376.3 + 376.3 - 376.3$ $+ 10.75 - 75.3 - 10.75)^2 \times (.001)^2$	1.11
$C_{\delta_2} = (215.2 - 193.6 - 752.6)^2 \times (.02)^2$	213.74
$C_{\epsilon_1} = (376.3)^2 + (376.3)^2 + (376.3)^2 + (376.3)^2 +$ $(-376.3)^2 \times (.005)^2$	17.70
$C_{\epsilon_2} = 4(53.8)^2 + 4(-48.4)^2 + (-376.3)^2 +$ $(-376.3)^2 \times (.03)^2$	273.74
$C_{\epsilon_3} = (10.75)^2 + (-75.3)^2 + (-10.75)^2$ $\times (.006)^2$.21
Sub-total.	<hr/> 506.5

Variance in sampling for element determination

$C_{\Delta_1} = (752.6 + 752.6 - 376.3)^2$ $\times (.02)^2$	509.77
$C_{\Delta_2} = (215.2 - 193.6 + 10.75 - 75.3 - 10.75$ $- 752.6 + 430.1 - 322.6 + 1032.3 - 1032.3)^2$ $\times (.006)^2$	17.58
$C_{\mu_1} = (752.6 + 752.6 - 376.3 + 215.2 - 193.6$ $+ 10.75 - 75.3 - 10.75 + 430.1 - 322.6$ $+ 1032.3 - 1032.3)^2 \div 58 \times (.01)^2$	2.41
$C_{\mu_2} = (-376.3)^2$ $\div 2 \times (.07)^2$	346.92
Sub-total	<hr/> 876.68

Variance in element analysis determination

$C_{\delta 1} = (752.6 + 752.6 - 376.3 + 215.2 - 193.6$ $+ 10.75 - 75.3 + 10.75 + 430.1 - 322.6$ $+ 1032.3 - 376.3 - 376.3 - 1032.3)^2$ $\times (.004)^2$	3.26
$C_{w1} = (752.6 + 752.6 - 376.3 + 215.2 - 193.6$ $+ 10.75 - 75.3 - 10.75 + 430.1 - 322.6$ $+ 1032.3 - 1032.3)^2 \div 58 \times (.01)^2$	2.41
$C_{w2} = (-376.3 - 376.3)^2 \div 2 \times (.07)^2$	1387.70
Sub-total	<u>1393.37</u>

Variance in measurements for isotope $C \sigma^2 \text{ (gm}^2\text{)}$

$C_{\delta 1} = (700 + 700 - 350 + 10 - 70 - 10)^2$ $\times (.001)^2$	0.96
$C_{\delta 2} = (200 - 180 - 700)^2 \times (.02)^2$	184.96
$C_{\epsilon 1} = 2(350)^2 + 2(350)^2 + (-350)^2$ $\times (.005)^2$	15.31
$C_{\epsilon 2} = 4(50)^2 + 4(-45)^2 + 2(-350)^2$ $\times (.03)^2$	236.79
$C_{\epsilon 3} = (10)^2 + (-70)^2 + (-10)^2$ $\times (.006)^2$	0.18
Sub-total	<u>438.2</u>

Variance in sampling for isotope

$C_{\delta 1} = (700 + 700 - 350 + 200 - 180$ $+ 10 - 75 - 10 - 700)^2 \times (.02)^2$	34.81
$C_{u1} = (700 + 700 - 350 + 200 - 180$ $+ 10 - 75 - 10)^2 \div 16 \times (.01)^2$	6.19
$C_{u2} = (-700)^2 \div 2 \times (.07)^2$	1200.5
Sub-total	<u>1241.5</u>

Variance in analysis for isotope determinationC σ^2 (gm²)

$Cv_1 = (700 + 700 - 350 + 200 - 180 + 10 - 75 - 10 - 700)^2$	$\times (.009)^2$	7.05
$Cv_2 = (400 - 300 + 960 - 960)^2$	$\times (.04)^2$	16.0
$Cv_1 = (1400 - 350 + 200 - 180 + 10 - 75 - 10 + 960 - 700)^2$	$\div 18 \times (.045)^2$	177.19
$Cv_2 = (400 - 300 + 960 - 960)^2$	$\div 166 \times (.04)^2$.10
	Sub-total	<u>200.34</u>

Summary of variance components for element determination

<u>Operation</u>	<u>Error Type</u>	<u>Variance</u>
Volume Measurement	LTS + R	506.5
Sampling	LTS + R	876.68
Analysis	LTS + R	393.37
	TOTAL	<u>2776.55</u>
	LEMUF =	$2(2776.55)^{\frac{1}{2}} = 105.4$ gms

Summary of variance components for isotope determination

<u>Operation</u>	<u>Error Type</u>	<u>Variance</u>
Volume Measurement	LTS + R	438.2
Sampling	LTS + R	1241.5
Analysis	LTS + R	200.34
	TOTAL	<u>1880.04</u>
	LEMUF =	$2(1880.04)^{\frac{1}{2}} = 86.7$ gms

APPENDIX F

DETERMINATION OF URANIUM IN ELECTROLYTE SOLUTIONS

SCOPE

This method is designed for the determination of total uranium in the electrolyte solutions used in the ^{235}U target production.

PRINCIPLE OF METHOD

An aliquot of the solution is fumed with H_2SO_4 , the uranium is reduced in the Jones Reductor and titrated with KMnO_4 .

SPECIAL APPARATUS AND REAGENTS

Nine-inch Jones Reductor - Place a perforated porcelain plate in the bottom of the reductor tube, followed by a small wad of glass wool. Fill to the neck with amalgamated zinc. Prepare the zinc as follows: shake 800 g. of 20 to 30 mesh zinc with 400 ml of HgCl_2 (25 g. per liter) in a liter flask for 2 minutes. Wash several times with H_2SO_4 (5 + 95) and then thoroughly with water. Keep the reductor filled with water when not in use.

0.1N POTASSIUM PERMANGANATE

Stock Solution - Dissolve 6.25 g. of KMnO_4 in 50 ml of boiling water.

While still hot, filter through glass wool into a 100 ml volumetric flask and dilute to volume.

0.05N KMnO_4 - Filter 27.0 ml of stock solution into a 1000 ml volumetric flask through burned off asbestos and dilute to volume.

Standardization - Weigh 0.3000 g. of sodium oxalate (N.B.S.) into a 600 ml beaker. Add 250 ml of H_2SO_4 (5 + 95) which has been boiled and cooled to 27°C . Stir until dissolved and add 39 to 40 ml of KMnO_4 solution. Stir slowly and allow to stand until the pink color disappears. Heat to 55 to 60°C and complete titration at this temperature. The end point should remain for 30 seconds. Determine a "blank" using the same volume of H_2SO_4 (5 + 95) and subtract. Calculate the normality, adjust to 0.1N KMnO_4 with water, and restandardize.

(0.300 g. of sodium oxalate (N.B.S.) is equivalent to 44.78 ml of 0.1N KMnO_4 or 22.39 ml of 0.05N KMnO_4 .)

APPENDIX F (cont'd)

PROCEDURE

Take a 10 ml aliquot of the electrolyte solution and transfer it to a 400 ml beaker, add 12 ml of 1.1 H₂SO₄, cover with watch glass and heat till fumes of SO₃ evolve. Cool the beaker, wash down the sides and watch glass, cover with water and refume to strong SO₃ fumes.

Adjust the volume of the uranium solution to about 100 ml and warm on the hot plate. Add KMnO₄ solution (25 g/l) dropwise until a pink color persists. Cool to room temperature.

Prepare the Jones Reductor by passing 100 ml of H₂SO₄ (5 + 95), followed by 100 ml of water through it. Discard these solutions. Pass the uranium solution through the reductor into the flask receiver. Wash the reductor with 100 ml of cold H₂SO₄ (5 + 95). Blow clean air through the reduced solution for 5 minutes. Wash the air purge column with water, allow to run into receiver, and remove the receiver from the flask. Titrate the solution in the receiver with standardized KMnO₄.

Run a blank using all things except uranium, and correct the volume of titrant for this blank.

CALCULATIONS

$$\frac{(A - B) \times 0.119 \times N (\text{KMnO}_4) \times 1000}{W} = \frac{\text{g/l}}{U}$$

CODE:

A = volume of titrant used

B = volume of titrant used for blank determination

N = normality of KMnO₄ solution (standardized by the analytical lab)

W = volume of aliquot taken by pipette

APPENDIX G

RADIOCHEMICAL SEPARATION FOR URANIUM

Reagents:

- 1) 5% Tri-n-octylamine V/V in Petroleum ether with 2%-Octyl alcohol added.
- 2) M-cresol purple indicator, ~ 0.04 gm dissolved in 100 ml of demineralized water.
- 3) Blast burner or hot plate, centrifuge cones, separatory funnels.

Separation:

- 1) Obtain sample from Hot Lab, be sure total volume and batch number are on paper work.
- 2) Do analyses in duplicate, take a sample split and place in a 250 ml Vycor beaker.
- 3) Add 1 ml con. HNO_3 , 1 ml HCl , and 1 ml HBr , and fume down to white fumes of H_2SO_4 (H_2SO_4 is waste solution).
- 4) If charring shows, add 1 ml of con. HNO_3 and repeat until no charring occurs.
- 5) When cool, add 100 ml of water and several drops of m-cresol indicator. If at proper pH, indicator will turn red.
- 6) Wash solution into a 250 ml separatory funnel having 10 ml of 5% TOA. Rinse beaker with ~ 100 ml of H_2O and put into separatory funnel.
- 7) Using wrist shakers, shake funnel for ~ 1 minute, allow to stand and draw off aqueous layer and discard. Add 25-30 ml of 0.1 N- H_2SO_4 and shake ~ 15 seconds to wash out organic layer. Allow to stand and draw off aqueous layer and discard.
- 8) Draw off organic layer into a calibrated centrifuge cone and spin for 2-3 minutes. Measure volume of organic layer.
- 9) For nondestructive counting draw off 3 ml of solution and count for ^{235}U , adjusting for fraction of total volume.
- 10) For chemical analysis of Total Uranium, draw off 10 ml of organic (or more) and do chemical analysis for total uranium.

NOTE: The extraction of Uranium VI at pH of 1.0-1.4 is quantitative but each transfer must also be quantitative.

²³⁵U ASSAY ON LIQUID SAMPLES

(QUALITY CONTROL LAB)

These liquid samples may be from stock plating solutions, plating waste, and fission waste, as well as liquid standards.

1. Obtain bulk tap of sample from processing group.
2. Count ¹³⁷Cs (NBS #82) check source on #2 NaI system at 10 cm for 1-5 minutes; read out results. Do this at the beginning and end of assay period; record results in log book.
3. Count ²³⁵U flame sealed vial 1 on base for 5 minutes at the beginning, middle, and end of the assay period. Record results in log book.
4. From the bulk tap of the sample, take duplicate aliquots (10 λ to 3 ml depending on uranium concentration) and place in standard disposable plastic vials and adjust to a 3 ml volume. Label vials with their sample code and aliquot and count on base for 5 minutes. Note: the integrated photo peak area should be in the 10,000 c/5 minute area to obtain 1% counting statistics. If the count rate is significantly below this (< 5000 counts), take a larger aliquot if possible. (Note 1)
5. Log all results in the log book; take average value of c/5 m for calculation of gms of ²³⁵U. If the difference between the two counts (splits) is greater than 5%, redo analysis using two more splits. If the difference can not be resolved, notify Q.A.
6. Compute the gms of ²³⁵U per ml of solution using the formula below.

$$\frac{(c/5 \text{ m}) \text{ Photo Peak Area} \times \text{dilution factor} \left(\frac{1 \text{ ml}}{\text{split size}} \right)}{3.0658 \times 10^6 \text{ (standard c/5 m/gm } ^{235}\text{U)}} = \text{gms } ^{235}\text{U}$$

7. Plot the average value of the ^{235}U control standard plus its 1σ spread on the control chart to assure the equipment is functioning correctly. If the average value falls outside the control value, hold the analysis and notify Q.A. Review data to ascertain reason for difference.
8. Report results out to the proper department.
9. Once a month, review results with Q.A.; be sure all control charts are kept up to date.

Note 1 - For the fission waste sample - this will be from an extraction for uranium in n-octyl amine. To dilute to 3 ml if necessary, you must use petroleum ether and not 1:5 HNO_3 as in the inorganic aqueous samples.

12/74

URANIUM-235 TARGET ASSAY PROCEDURE

(QC Lab)

1. Obtain target cylinder after Health Physics has certified the outside is clean.
2. Count ^{137}Cs NBS #82 check source in #2 NaI system 10 cm for 1-5 minutes; read out on tally, typewriter or other and record.
3. Count whole cylinder on top of geometry stand (32 cm) making sure it is centered. Count 0-1 MeV/400 ch for 5 minutes and read out first 100 channels on tally, typewriter or other.
4. Submit spectra to the NUMPLA computer program or compute by hand.
5. Calculate total gms of ^{235}U present using photopeak area of gamma at channel 75 (185 keV gamma of 7.1×10^8 year ^{235}U). Gamma ray is 54% relative intensity.
6. Count standard tube at 32 cm geometry 3 times during counting sequence; record data and plot on control chart.

Calculations:

$$\frac{\text{Photopeak area (c/5 m)}}{A \times B \times C} = \text{gms } ^{235}\text{U}$$

A - ^{137}Cs c/m on calibration date 11/74 was 163,113 c/5 m \pm 0.5%.

B - Counts-per-minute-per-gram of ^{235}U from several calibrated sources 11/74 was 17,391 c/5 m/gm.

C - The standard tube #804 was 166,518 c/5 m on 11/74, its control limits are \pm 5%.

Note: If either standard does not fall within its stated deviation, notify Quality Assurance.

11/74 revised