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DOCUMENT NO.

HW-58278

SERIES AND COPY NO.

MB-1

GENERAL ELECTRIC

HANFORD ATOMIC PRODUCTS OPERATION – RICHLAND, WASHINGTON

DATE

NOVEMBER 25, 1958

[redacted]

TITLE

REDOX ACCOUNTABILITY TEST PROGRAM – INITIAL RESULTS

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Verified By J E Savely 2-7-94

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REDOX ACCOUNTABILITY TEST PROGRAM - INITIAL RESULTS

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REDOX ACCOUNTABILITY TEST PROGRAM - INITIAL RESULTS

I. INTRODUCTION

In an effort to resolve present 100 Area-200 Area uranium and plutonium B-PID's, a large scale accountability test program was recently carried out in the Redox Facility. The test, as originally planned, was to consist of the complete processing (no inventory-clean plant basis) of about 55 tons of selected metal in conjunction with an extensive analytical, sampling, and volume measurement program designed to provide the following fundamental data:

1. A comparison of 100 Area predicted plutonium values versus a) H-7 tank receipts, b) L-6 load-outs, and c) plutonium input values calculated by the plutonium-to-uranium ratio method.
2. A comparison of 100 Area uranium values versus H-7 tank receipts and E-12 tank load-outs.
3. A complete material balance for uranium and plutonium through the Redox plant, which in turn will provide a comparison of measured waste losses versus receipts minus total product recovered.
4. A measure of sampling precision and accuracy.
5. An estimate of volume measurement accuracy at H-7 and E-12.
6. A test for possible inherent analytical biases (method biases) that are not detected by normal quality control standards.
7. An evaluation of the application of analytical bias corrections based on quality control data.

With the exception of two incidents, the processing requirements (minimum inventory and measurement of all material) necessary to the success of the test, were met. The two incidents which increase the uncertainties associated with some of the material balance values obtained were: 1) the discharge of an estimated 700 pounds of uranium to the floor in a transfer from F-5 to F-4 due to the improper installation of the F-5 to F-4 transfer line (jumper) and 2) the discovery of a large accumulation of plutonium ($\sim$15 kg) in the L-2 stripping tower after completion of the test run. The first incident decreases the accuracy which can be attached to E-12 load-out values, whereas the second incident decreases the accuracy which can be attached to L-6 load-out values. However, from the data obtained in the pre-processing and post-processing flushes, it is generally believed that the material found in the L-2 stripping tower represents a long term accumulation and neither contributed to L-6 load-out values nor represents a part of the test runs.

An important qualification in regard to the 100 Area-200 Area test relationship is the fact that the test material was from the old piles (D pile, H pile, and DR pile) and was of relatively low average MWD/T, $\sim$ 500. Consequently, it is more representative of 100-Area production prior to receipt of production from the K-piles than of recent (FY-57 and 58) production. It should also be noted that the test run did include one small (~2 tons) batch of high MWD/T (~[illegible]00 MWD/T) from DR pile which was dissolved and sampled separately before being combined with the rest of the test material.

Excluding the above qualifications, the material balances and other pertinent measurement data obtained can be considered as representative of typical plant operation and nuclear material control.

The purpose of this report is to present the measurement data (obtained to date) for further study and analysis and to summarize those conclusions which can be justified at this time.

II. SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

A. Summary

The processing phase of the test run and all plant and control laboratory measurements have been completed. This data is summarized and discussed in this report. Process Chemistry measurements, which include measurement of the H-7 plutonium-to-uranium ratio (on the H-7 composite) by independent analytical methods, measurement of plutonium and uranium isotopic concentrations, and measurement of the special batch of high level MWD/T, have not been completed and will be reported separately.

B. Conclusions

The conclusions listed below are based on preliminary examinations of the measurement data of more immediate concern. More complete presentation of the significance of the measurement data will be forthcoming from the various specialists concerned.

1. In the test run, excellent agreement was achieved between plutonium in-put values, as calculated by the plutonium-to-uranium ratio method, and load-out values. This close agreement reinforces the view that CPD in-put, waste, and load-out measurements for plutonium are reliable.

2. For this lot of material, Redox plutonium values at H-7 and L-6 were significantly higher (3-5%) than the 100 Area predicted values. This illustrates the difficulty in making accurate exposure calculations for small batches of material and lends weight to the argument that 100 Area predicted values or small batches are not a reliable measure of the plutonium content. It also seemingly refutes the view that current 100 Area values are consistently over-stated. It should be noted, however, that the material used in this run was all produced in the old piles. We believe that the predicted values for the K-pile material, which made up about 40 per cent of the production for the last two years, are more likely to be over-stated than are the values for the older piles. In consequence of these factors, and in view of the apparent reliability of CPD measurements, which have shown general 100 Area over-statement over the past two years, we do not believe that the plutonium recovery on this run refutes the probability of general continued 100 Area over-statement.

3. Individual plutonium-to-uranium ratios which are the quotient of two independent measurements appear to be as precise as the individual plutonium values. This lends support to the view that the ratio method is less subject to sampling variation than the volume-concentration method.

4. Sampling is not a major source of concern at H-7, E-12, and L-[illegible]

5. H-7 uranium values (corrected for analytical biases) determined by the conventional density method (UD-1a) are consistently higher than corresponding values obtained by the TBP Extraction-X-ray Absorption (UX-4a) method. However, further work is needed to establish the exact cause and magnitude of this discrepancy.

6. D-[illegible] and H-10 measurements have the largest variance of all the accountability measurements made. However, this variance does not appear to be due to consistent errors and because of the small amount of material involved these measurements are not a major source of concern

C. Recommendations

The following future actions are recommended:

1. The plutonium-to-uranium ratio, as measured at H-7 at Redox and at E-5 at Purex, should be used routinely to correlate 100 Area-200 Area plutonium values. Future accountability tests used to establish this relationship should be designed to take advantage of the routine application of the ratio method.

2. A thorough statistical analysis of the measurement data should be made to establish sampling precision and accuracy. These values should then be presented to the Atomic Energy Commission as evidence of the Department's performance and efforts in this regard.

3. Analytical bias corrections should be applied to the H-7 uranium, plutonium, and specific gravity values. The H-7 plutonium correction should include bias corrections for americium-curium in addition to those applied to the "F" factor and the total alpha measurement.

4. Analytical tests designed to uncover inherent analytical biases (those biases which are due primarily to process constituents such as corrosion products, fission products, etc., and are not detected by control standards) should be continued on the H-7 specific gravity uranium method, the L-6 plutonium X-ray method, and the H-7 americium-curium method. When a definite inherent bias has been established it should be applied to source data.

III. DISCUSSION

A. Introduction

[illegible] the primary purpose of the test run was to make a comparison of 100 Area [illegible] values versus 200 Area measured values using a selected batch of metal,

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the bulk of extraneous material such as dissolver heels and normal process hold-up material was processed and removed prior to the test. In addition, to meet material balance requirements of the test, all process vessels were flushed prior to the test and the SS materials accumulated in the flushes (beginning inventory) were assayed by the most accurate techniques practical. Following plant clean-out and measurement of the beginning inventory (which amounted to less than 1% of the run material), the test metal was dissolved and processed on a complete recovery and complete measurement basis. In order to obtain the highest precision and accuracy practical, and also to establish experimental evidence of the validity of plant measurements, an extensive analytical, sampling, and volume measurement program was carried out. The material balances obtained and various experimental phases of the test program are discussed separately in the succeeding sections.

B. Uranium Material Balance

The uranium material balance obtained and the amount of material involved in beginning and ending inventory are illustrated in Tables I and III. The individual H-7 and E-12 batch sizes and the magnitude of the analytical bias corrections applied are listed in Tables IV and V.

As is shown by the data in Table I, adjusted H-7 uranium values agree within 0.5 - 2.1% of adjusted E-12 load-out values, depending on the analytical method used at H-7 and the application of analytical bias corrections. The application of analytical bias corrections increases the discrepancy between H-7 and E-12. This discrepancy is further increased by the use of the TBP Extraction-X-ray Absorption method (UX-4a) in place of the conventional Density-Calculation method (UD-1a) for uranium. In the case of these two analytical methods, the application of analytical bias corrections decreases the between-method discrepancy.

The general agreement between the three uranium balance areas H-7, E-12, and 100-Area eliminates the possibility of large undetected losses. However, because of the uncertainties involved in the measured values, it does not eliminate the possibility of small undetected losses.

The fact that the corrected H-7 values are lower than both the 100 Area and E-12 values indicates the possibility of a volume measurement bias at H-7. However, whether a significant bias exists or not is dependent on a further statistical analysis.

As yet, the physical cause and the exact magnitude of the between-method bias (UD-1a versus UX-4a) has not been fully determined. Since the Density-Calculation method is "blind", i.e., it reports anything that contributes to the density of the solution as uranium, it is believed that part of this discrepancy is due to the presence of small amounts of process contaminates in the H-7. The most likely source of such contaminates is from the traces of coating waste solution not completely removed from the dissolver prior to the acid addition and metal dissolution step. Although it is generally felt that the amount of bias due to this type of effect is small (< 0.[illegible]%), it is still necessary to establish its magnitude and constancy in order to make a valid bias correction.

C. Plutonium Material Balance

The plutonium material balance obtained and amount of material involved in beginning and ending inventory are shown in Tables II and III. The individual batch sizes and the magnitude of the analytical bias correction applied are shown in Table VI.

As shown by the data in Table II, Redox plutonium values at all measurement points and by all the various methods used are significantly higher (~[illegible]%) than 100 Area predicted values. In view of the run size (~55 tons) and the normal uncertainties in predicted values, this disparity between 100-Area and 200-Area plutonium values is not surprising. Also, it should be noted that the material is entirely from the old piles and as such is not representative of average current production.

The most significant aspect of the plutonium balance is the degree of agreement between H-7 and L-6 values by the various methods. As is illustrated in Table II, the conventional H-7 measurements are about 1.5% lower than L-6 load-out values, with the difference being only slightly affected by the application of analytical bias corrections. The best agreement between H-7 and L-6 values is obtained by applying the H-7 plutonium-to-uranium ratio to 100 Area predicted values, with the best agreement being obtained when the X-ray uranium (UX-4a) values are used. This result lends further support to the possibility of an inherent high bias in the conventional Density-Calculation method.

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TABLE I

PLANT URANIUM BALANCE

Measurement Area		Pounds Uranium	% BPID Redox - 100 Area
100 Area Value		111,134	
Redox Values			
1) H-7 Receipts by UD-1a(1) + coating Waste Losses	(3) Corrected	110,197	-0.84
	Uncorrected	111,068	-0.06
2) H-7 Receipts by UX-4a(2) + coating Waste Losses	Corrected	109,359	-1.60
	Uncorrected	109,469	-1.50
3) E-12 load-outs corrected for waste losses and inventory	Corrected	111,766	+0.57
	Uncorrected	111,655	+0.47

(1) Uranium as determined by the conventional laboratory specific gravity method in which uranium is calculated from the H-7 laboratory specific gravity.

(2) Uranium as determined by the TBP Extraction-X-ray Absorption method.

(3) Values corrected for analytical biases based on control standard recoveries.

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TABLE II

PLANT PLUTONIUM BALANCE

Measurement Area		Grams Plutonium	% BPID Redox - 100 Area
100 Area		23,839	
Redox Values			
1) H-7 Receipts by volume-concentration method plus coating waste losses	corrected[(1)]	24,596	3.2
	uncorrected	24,747	3.8
2) H-7 Receipts by Pu/U Ratio method using UD-1A Uranium[(2)] values	corrected	24,852	4.3
	uncorrected	24,808	4.1
3) H-7 Receipts by Pu/U Ratio method using UX-4a Uranium[(3)] values.	corrected	25,048	5.1
	uncorrected	25,172	5.6
4) L-6 Load-outs corrected for Waste Losses and inventory	corrected	24,975	4.8
	uncorrected	25,099	5.3

(1) Values corrected for analytical biases based on control standard recoveries.

(2) Uranium as determined by the conventional laboratory specific gravity method in which uranium is calculated from the H-7 laboratory specific gravity.

(3) Uranium as determined by the TBP Extraction-X-ray Absorption method.

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TABLE III

BEGINNING AND ENDING INVENTORY AND WASTE LOSSES

Account	Pu-grams	U-pounds	
Beginning Inventory (H-4 tank)	199.1	353.3	
Coating Waste losses	40.3	388.0	
D-9 Waste losses corrected for D-1	94.4	111.3	
Ending Inventory (H-4 Tank)	184.1	323.7	
Amount added to H-7 Receipts	40.3	388.0	
Amount added to E-12 Load-out values		682.6	709 (1)
Amount Added to Pr Load-out values	119.7		

(1) 709 lbs. (estimated) of U lost to floor during transfer from F-5 to F-4 because of an improperly installed jumper.

TABLE IV

H-7 RECEIPTS

RAW DATA

Batch No.	Pounds Uranium By UD-1a (1)	Pounds Uranium By UX-4a (2)	Grams Plutonium (3)
MS-30	7934.2	7843.3	1867.1
MS-31	7927.5	7827.5	1884.9
MS-32	7961.1	7896.5	1856.8
MS-33	7872.3	7749.1	1745.9
1	8105.4	7925.6	1851.9
2	8032.4	7924.2	1753.9
3	15,974.6	15,839.8	3376.3
4	8179.9	8019.5	1833.7
5	16,345.5	16,137.4	3141.0
6	7960.5	7766.5	1495.5
7	7961.6	7800.8	1738.2
8	5927.0	5852.2	1159.3
9	488.5	488.5	55.3
AF-1	5.8	5.8	2.1
AF-2	3.4	3.4	2.6
Total	110,679.7	109,080.7	23,764.5
Coating Waste	388	388	40.3
"F" Factor Correction			+ 942
Analytical Bias correction	- 871	- 110	- 151
Corrected total	110,197	109,359	24,596
Uncorrected total	111,068	109,469	24,747

(1) Uranium calculated from laboratory specific gravity.

(2) Uranium by TBP Extraction-X-ray-Absorption method.

(3) Plutonium by Radioassay

[redacted]

TABLE V

E-12 LOAD-OUTS

Batch No.		Pounds Uranium[1]
1		6328.4
2		6589.7
3		5424.5
4		6527.7
5		6297.8
6		6329.3
7		6718.5
8		6793.3
9		7077.6
10		7409.1
11		6785.3
12		7843.7
13		6706.3
14		6576.1
15		6814.7
16		6868.7
17		2780.1
18	Flush	1.6
	Uncorrected total	110,972.4
	Analytical bias correction	+ 111
	Corrected total	111,083

(1) Values determined by X-ray Absorption method.

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TABLE VI

L-6 LOAD-OUT VALUES

Run No.	Can No.	Grams Plutonium by PUX-3a(1)	By PUA-6b(2)	
1	S-106	1446.3		
	A-37	1453.0		
	A-28	1466.6		
2	A-41	1749.6		
	A-25	1758.6		
	S-31	1777.5		
3	A-20	1870.5		
	A-24	1854.7		
	A-12	1859.9		
4	S-136	1858.1		
	S-134	1861.2		
	S-129	1687.2		
5	A-14	1289.9		
	A-19	1266.3		
6	S-122	441.8		
	S-127	465.2		
7	S-124, S-101, A-47, A-45		371.8	
8	A-24, A-6, A-20, S-123		161.0	
9	S-138, A-28		120.3	
10	L-6 Flush		134.6	
11	L-6 Flush		85.1	
	Uncorrected Sub-totals	24,106.4	872.8	
	Uncorrected totals			24,979.2
	Analytical Bias Correction	-119.9	-4.3	
	Corrected total	23,986.5	868.5	24,855.0

(1) Plutonium by X-ray Absorption

(2) Plutonium by Radioassay using an "F" of 1.347 based on average of R-5 and R-6.

[redacted]

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D. Measured Waste Losses

The waste losses sustained in the test run are illustrated in Table VII and the individual analytical values are listed in Tables XV and XVI, page [illegible] and [illegible].

As is demonstrated in Table VII, the largest single waste loss occurred in a coating waste batch (C-1 batch). Since this happening is the result of a process error (the inadvertent jetting of the dissolver rinse solution to the coating waste tank), it can not be considered as representative of normal plant operation. More important than loss itself is the fact that large coating waste losses are readily detected, a fact which has long been the subject of dispute due to the chemical and physical nature of coating waste.

TABLE VII

MEASURED WASTE LOSSES

Measurement Point	Batch No.		Grams Plutonium (1)		Uranium Pounds (1)
H-10	C-1		32.7		347.2
	18		0.7		1.4
	19		1.0		3.5
	20		1.2		4.9
	D-2		2.[illegible]		18.8
	D-4		2.0		12.2
		Total	40.3	Total	388.0
D-2	Composite		<[illegible]		<1
D-9	PW-2		7.9		11.4
	PW-3		4.7		19.5
	PW-4		9.6		12.6
	PW-5		18.0		47.2
	PW-6		8.8		25.3
	PW-7		9.4		31.6
	PW-8		8.8		27.0
	PW-9		4.4		12.3
	PW-11		9.2		20.[illegible]
	PW-12		10.6		11.8
	PW-13		5.5		4.4
	PW-14		3.4		1.9
	PW-15		5.8		3.0
	PW-16		9.8		8.5
	PW-17		8.5		26.1
		Total	124.4	Total	263.0
			- 30.0		- 151.7
D-9 - D-1 (2)		Total	94.4	Total	111.[illegible]

(1) All uncorrected for analytical biases and "F" factor adjustment.
(2) D-9 waste included 30 grams of plutonium and 151.7 pounds of uranium from D-1 which was present before Special Run.

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E. Manometer and Scale Checks

In an effort to estimate the precision of plant manometer readings and FR-can weighings, the following special tests were made: 1) two manometer readings taken 5 minutes apart were made on each H-7 and E-12 volume measurement and 2) with each FR-can weighing a comparison was made of the observed tare weight and observed tare weight plus 10 kilogram check weight. In all cases, the two manometer readings were identical. In the weighing tests, the maximum difference observed between the tare weight and the tare weight plus 10 kilogram check weight was 25 grams, with the average deviation for the 21 weighing tests made equal to (+) 8 grams.

F. Sampling and Analytical Precision

The raw data (uncorrected for analytical biases) illustrating sampling and analytical precision at the various accountability points is given in the following tables:

1. H-7 Uranium Values, Table VIII, page 16.
2. H-7 Plutonium Values, Table IX, page 18.
3. H-7 Plutonium to Uranium Ratios, Table X, page 20.
4. E-12 Uranium Values, Table XI, page 22.
5. L-6 Plutonium and "F" Factor Values, Table XII, page 24.
6. H-7, E-12, and L-6 Incidental Analysis, Table XIII, page 27.
7. D-9 Plutonium and Uranium Values, Table XIV, page 28.
8. H-10 Plutonium and Uranium Values, Table XV, page 30.

The most significant fact illustrated by the experimental data is the excellent sampling and analytical precision obtained at H-7, E-12, and L-6. From the standard deviations, which include both sampling and analytical variance of the individual results, it is evident that sampling is not a major source of variation at H-7, E-12, and L-6.

Another item of importance is the comparatively high precision of the individual plutonium to uranium ratios illustrated in Table X. Since the ratio includes two independent measurements, uranium and plutonium, it would be reasonable to expect a larger variance for the ratios than for either of the individual measurement variances. However, as the data shows, the precision of the individual ratios compares very favorably with the precision of the individual plutonium values (S = 1.83% for individual Pu results on same run versus S = 1.91% for R using UX-4a uranium and S = 1.45% for R using UD-1a uranium). Since "S" includes sampling variance, this result reinforces the view that the ratio method is less subject to sampling variations and other extraneous effects [illegible] the present volume concentration system.

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TABLE VIII

H-7 URANIUM ANALYTICAL VALUES[1]

Run No.	Sample No.	Method: UD-1a[2] Original lbs/gal-U	UD-1a[2] Referee lbs./gal-U	UX-4a[3] Original lbs/gal-U	UX-4a[3] Referee lbs/gal-U
MS-30	1[4]	4.12	4.13	4.06	4.05
	2	4.16	4.13	4.12	4.06
	3	4.13	4.13	4.11	4.12 RR[5]
					4.07
	4	4.13	4.13	4.05	4.04 RR
					4.18
	5	4.19	4.19	4.15	4.15
		$\bar{X}$ = 4.1440		$\bar{X}$ = 4.0967	4.25 RR
MS-31	1	4.27	4.26	4.28	4.20
	2	4.26	4.27	4.15	4.17
	3	4.25	4.29	4.20	4.21
		$\bar{X}$ = 4.2667		$\bar{X}$ = 4.2128	
MS-32	1	4.52	4.52	4.53	4.47
	2	4.51	4.55	4.53	4.49
	3	4.52	4.52	4.46	4.44
		$\bar{X}$ = 4.5233		$\bar{X}$ = 4.4867	
MS-33	1	4.49	4.48	4.45	4.41
	2	4.49	4.50	4.47 RR	4.43
	3	4.50	4.50	4.45	4.45
		$\bar{X}$ = 4.4933		$\bar{X}$ = 4.4433	
MS-1	1	4.49	4.46	4.35	4.26
	2	4.52	4.53	4.50	4.37
	3	4.52	4.53	4.50	4.47
		$\bar{X}$ = 4.5083		$\bar{X}$ = 4.4083	
MS-2	1	4.52	4.54	4.35	4.34
	2	4.55	4.55	4.51	[illegible]
	3	4.59	4.51	4.57	4.52
		$\bar{X}$ = 4.5433		$\bar{X}$ = 4.4817	
MS-3	1	4.37	4.37	4.33	--
	2	4.37	4.38	4.31	4.26
	3	4.41	4.41	4.44	4.40
		$\bar{X}$ = 4.3850		$\bar{X}$ = 4.3480	

[redacted]

[redacted]

TABLE VIII (Cont.)

		Method			
		UD-1a		UX-4a	
Run No.	Sample No.	Original lbs/gal-U	Referee lbs/gal-U	Original lbs/gal-U	Referee lbs/gal-U
MS-4	1	4.35	--	4.35	--
	2	4.36	4.41	4.30	4.2[illegible]
	3	4.39	4.3[illegible]	4.26	4.31
		$\bar{X}$ = 4.3860		$\bar{X}$ = 4.3000	
MS-5	1	4.48	4.54	4.39	4.47
	2	4.48	4.47	4.43	4.47
	3	4.46	4.43	4.33	4.4[illegible]
		$\bar{X}$ = 4.4767		$\bar{X}$ = 4.4200	
MS-6	1	4.35	4.30	4.29	4.25
	2	4.38	--	4.2[illegible]	--
	3	4.34	4.38	4.26	4.22
		$\bar{X}$ = 4.3500		$\bar{X}$ = 4.2440	
MS-7	1	3.42	3.40	3.41	3.32
	2	3.41	3.43	3.36	3.36
	3	3.42	3.42	3.28	3.36
		$\bar{X}$ = 3.4167		$\bar{X}$ = 3.3483	
MS-8	1	3.11	3.08	3.05	3.11
	2	3.10	3.08	3.06	3.02
	3	3.09	3.06	3.02	3.03
		$\bar{X}$ = 3.0867		$\bar{X}$ = 3.0483	
		$\bar{\bar{X}}$ = 4.215	$\bar{\bar{S}}$ = 0.557%(6)	$\bar{\bar{X}}$ = 4.153	$\bar{\bar{S}}$ = 1.38%

(1) Raw data uncorrected for analytical biases.

(2) Uranium calculated from the H-7 laboratory specific gravity.

(3) Uranium by the TBP Extraction-X-ray Absorption method.

(4) Samples taken eight minutes apart with full sampler circulation between samples and continuous tank agitation.

(5) RR = analytical reruns.

(6) The standard deviation of an individual analytical result on the same run - obtained from the pooled variance and includes sampling and analytical variance but not between-run variance.

[redacted]

TABLE IX

H-7 PLUTONIUM ANALYTICAL VALUES

Run No.	Sample No.[3]	Plutonium Grams/Gallon[1] Original (PUA-5b)[2]	Referee
MS-30	1	0.970	
	2	0.970	
	3	0.986	0.976
	4	0.974	0.969
	5	0.984	0.978
		$\bar{X}$ = 0.97587	
MS-31	1	1.016	1.016
	2	1.015	1.021
	3	1.011	1.008
		$\bar{X}$ = 1.01450	
MS-32	1	1.040	1.016
	2	1.072	1.084
	3	1.043	1.076
		$\bar{X}$ = 1.05516	
MS-33	1	1.000	0.997
	2	0.983	1.010
	3	0.997	0.992
		$\bar{X}$ = 0.99650	
MS-1	1	1.030	1.030
	2	1.020	1.030
	3	1.040	1.030
		$\bar{X}$ = 1.03000	
MS-2	1	0.996	0.987
	2	0.996	0.984
	3	0.996	1.07
		$\bar{X}$ = 1.00483	
MS-3	1	0.934	0.908
	2	0.934	0.911
	3	0.937	0.937
		$\bar{X}$ = 0.92683	
MS-4	1	0.965	0.995
	2	0.970	0.981
	3	0.968	1.020
		$\bar{X}$ = 0.98316	
MS-5	1	0.883	0.861
	2	0.845	0.864
	3	0.845	0.864
		$\bar{X}$ = 0.86033	
MS-6	1	0.821	0.825
	2	0.813	--
	3	0.803	0.824
		$\bar{X}$ = 0.81720	

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TABLE IX (Cont.)

H-7 PLUTONIUM ANALYTICAL VALUES

Run No.	Sample No.	Plutonium Grams/Gallon Original (PUA-[illegible])	Referee
NS-7	1	0.771	0.75[illegible]
	2	0.740	0.744
	3	0.725	0.742
		$\bar{X}$ = 0.74600	
NS-8	1	0.602	0.610
	2	0.605	0.597
	3	0.600	0.60[illegible]
		$\bar{X}$ = 0.60383	
NS-9	1	0.0517	0.0505
	2	0.0474	--
	3	0.0466	0.0466
		$\bar{X}$ = 0.04856	
		$\bar{\bar{X}}$ = 0.865	$s^{(4)}$ = 1.83%

(1) Raw data uncorrected for analytical biases and not adjusted for "F" factor.

(2) Plutonium by direct radioassay.

(3) Sample taken eight minutes apart with full sampler circulation between samples and continuous tank agitation.

(4) The standard deviation of an individual analytical result on the same run - obtained from the pooled variance and includes sampling and analytical variance but not between-run (process) variance.

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TABLE X

H-7 PLUTONIUM TO URANIUM RATIOS

Run No.	Sample(5)	Grams Plutonium/Pound Uranium: Pu by PUA-6c(2), U by UX-4a(3) — Original	Referee	PU by PUA-6c, U by UD-1a — Original	Referee
MS-30	1	0.2389		0.2354	
	2	0.2354		0.2332	
	3	0.2399	0.2369 RR 0.2398	0.2357	0.2363
	4	0.2405	0.2398 RR 0.2318	0.2358	0.2346
	5	0.2371	0.2357	0.2348	0.2334
		$\bar{X}$ = 0.2376		$\bar{X}$ = 0.2353	
MS-31	1	0.2374	0.2391 RR 0.2419	0.2379	0.2385
	2	0.2428	0.2448	0.2383	0.2391
	3	0.2407	0.2394	0.2379	0.2350
		$\bar{X}$ = 0.2409		$\bar{X}$ = 0.2378	
MS-32	1	0.2296	0.2273	0.2301	0.2248
	2	0.2366	0.2414	0.2377	0.2382
	3	0.2338	0.2423	0.2308	0.2380
		$\bar{X}$ = 0.2352		$\bar{X}$ = 0.2332	
MS-33	1	0.2247	0.2261	0.2227	0.2225
	2	0.2199	0.2280	0.2189	0.2244
	3	0.2240	0.2229	0.2216	0.2204
		$\bar{X}$ = 0.2243		$\bar{X}$ = 0.2218	
MS-1	1	0.2368	0.2418	0.2294	0.2309
	2	0.2267	0.2357	0.2257	0.2274
	3	0.2311	0.2304	0.2301	0.2274
		$\bar{X}$ = 0.2338		$\bar{X}$ = 0.2285	
MS-2	1	0.2290	0.2274	0.2204	0.2174
	2	0.2208	0.2139	0.2189	0.2163
	3	0.2179	0.2367	0.2170	0.2372
		$\bar{X}$ = 0.2243		$\bar{X}$ = 0.2212	
MS-3	1	0.2157	--	0.2137	0.2078
	2	0.2167	0.2138	0.2137	0.2080
	3	0.2110	0.2130	0.2125	0.2125
		$\bar{X}$ = 0.2140		$\bar{X}$ = 0.2114	
MS-4	1	0.2218		0.2203	--
	2	0.2256	0.2292	0.2225	0.2224
	3	0.2272	0.2366	0.2205	0.2323
		$\bar{X}$ = 0.2281		$\bar{X}$ = 0.22[illegible]	

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TABLE X (Cont.)

Run No.	Sample(5)	Grams Plutonium Found Uranium: Pu by PUA-5b U by UX-4a Original	Referee	Pu by PUA-[illegible] U by UD-1a Original	Referee
MS-5	1	0.2011	[illegible]	[illegible]	[illegible]
	2	0.1907	0.1933	[illegible]	[illegible]
	3	0.1952	0.1950	[illegible]	[illegible]
		$\bar{X}$ = 0.1947		$\bar{X}$ = 0.1922	
MS-6	1	0.1914	0.1941	[illegible]	[illegible]
	2	0.1936	--	[illegible]	--
	3	0.1885	0.1953	[illegible]	[illegible]
		$\bar{X}$ = 0.1926		$\bar{X}$ = [illegible]	
MS-7	1	0.2261	0.2271	0.2254	[illegible]
	2	0.2202	0.2214	0.2170	[illegible]
	3	0.2210	0.2208	0.2120	[illegible]
		$\bar{X}$ = 0.2228		$\bar{X}$ = 0.2184	
MS-8	1	0.1974	0.1961	0.1935	0.1980
	2	0.1977	0.1977	0.1951	0.1938
	3	0.1987	0.2010	0.1942	0.1900
		$\bar{X}$ = 0.1981		$\bar{X}$ = 0.1956	
		$\bar{\bar{X}}$ = 0.2221		$\bar{\bar{X}}$ = 0.2180	
		$s^{(6)}$ = 1.91%		s = 1.45%	

(1) Raw data uncorrected for analytical biases. RR denotes analytical rerun.
(2) Plutonium by direct radioassay.
(3) Uranium by the TBP Extraction-X-ray Absorption method.
(4) Uranium calculated from the laboratory specific gravity.
(5) Samples taken 10 minutes apart with full sampler circulation between samples and continuous tank agitation.
(6) The standard deviation of an individual result on the same run - obtained from the pooled variance and includes sampling and analytical variance.

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TABLE XI

E-12 URANIUM ANALYTICAL VALUES(1)

Batch #	Sample No. (4)	Method: UD-1a (2) Original #/gal U	UD-1a (2) Referee #/gal U	UX-2a ([illegible]) Original #/gal U	UX-2a ([illegible]) Referee #/gal U
				3.45 RR(5)	
1	1	3.52	3.53	3.49	3.51
	2	3.48	3.48	3.45	3.45
		X̄ = 3.5025		X̄ = 3.4720	
2	1	4.11	4.12	4.09	4.10
	2	4.12	4.11	4.09	4.09
		X̄ = 4.1150		X̄ = 4.0925	
3	1	4.15	4.18	4.17	4.16
	2	4.15	4.18	4.15	4.14
		X̄ = 4.1650		X̄ = 4.1550	
4	1	4.18	4.19	4.15	4.16
	2	4.19	4.20	4.14	4.18
		X̄ = 4.1900		X̄ = 4.1575	
5	1	4.22	4.22	4.20	4.19
	2	4.22	4.22	4.20	4.20
		X̄ = 4.2200		X̄ = 4.1975	
6	1	4.20	4.20	4.20	4.19
	2	4.20	4.21	4.19	4.19
		X̄ = 4.2025		X̄ = 4.1925	
7	1	4.18	4.19	4.17	4.16
	2	4.19	4.19	4.18	4.19
		X̄ = 4.1875		X̄ = 4.1725	
8	1	4.16	4.17	4.16	4.1[illegible]
	2	4.16	4.17	4.15	4.15
		X̄ = 4.1650		X̄ = 4.1500	
9	1	4.16	4.17	4.14	4.13
	2	4.12	4.17	4.14	4.14
		X̄ = 4.1550		X̄ = 4.1375	
10	1	4.21	4.21	4.19	4.1[illegible]
	2	4.20	4.21	4.1[illegible]	4.1[illegible]
		X̄ = 4.2075		X̄ = 4.1800	

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TABLE XI (Cont.)

E-12 URANIUM ANALYTICAL VALUES

Batch #	Sample No.	Method: UD-1a (2) Original #/gal U	UD-1a (2) Referee #/gal U	UX-2a (3) Original #/gal U	UX-2a (3) Referee #/gal U
11	1	4.17	4.23	4.20	4.1[illegible]
	2	4.17	4.22	4.20	4.2[illegible]
		$\bar{X}$ = 4.1975		$\bar{X}$ = 4.2025	
12	1	4.21	4.21	4.18	4.19
	2	4.22	4.21	4.19	4.19
		$\bar{X}$ = 4.2125		$\bar{X}$ = 4.1875	
13	1	4.26	4.23	4.24	4.22
	2	4.26	4.23	4.24	4.23
		$\bar{X}$ = 4.2450		$\bar{X}$ = 4.2325	
14	1	4.24	4.26	4.21	4.22
	2	4.24	4.25	4.20	4.22
		$\bar{X}$ = 4.2475		$\bar{X}$ = 4.2125	
15	1	4.24	4.24	4.21	4.23
	2	4.24	4.25	4.24	4.23
		$\bar{X}$ = 4.2425		$\bar{X}$ = 4.2275	
16	1	4.17	4.17	4.14	4.13
	2	4.16	4.16	4.15	4.14
		$\bar{X}$ = 4.1650		$\bar{X}$ = 4.1400	
17	1	2.98	2.97	2.92	2.92
	2	2.94	2.97	2.94	2.93
		$\bar{X}$ = 2.9650		$\bar{X}$ = 2.9275	
		$\bar{\bar{X}}$ = 4.082		$\bar{\bar{X}}$ = 4.053	
		S = 0.294%(6)		S = 0.296%(3)	

(1) Raw data uncorrected for analytical biases.
(2) Uranium calculated from laboratory specific gravity
(3) Uranium by the Direct X-ray Absorption method.
(4) Samples taken 15 minutes apart with full sampler circulation between samples and continuous tank agitation.
(5) Analytical rerun.
(6) The standard deviation of an individual analytical result on the same batch - obtained from the pooled variance and includes sampling and analytical variance but not between (process) variance.

[redacted]

TABLE XII

L-6 PLUTONIUM ANALYTICAL VALUES(1)

Run No.	Sample No.	Sample Type(2)	PUX-3A(3) grams/gallon Original	PUX-3A(3) grams/gallon Referee	PUA-6B-1(4) counts/min/gallon Original	PUA-6B-1(4) counts/min/gallon Referee	F-Factor(5) grams/count/min. Original	F-Factor(5) grams/count/min. Referee
			Method					
R-1	111	L-6 Tank	657.2	658.4	5.01^{13}	5.00^{13}	1.312	1.317
	141	L-6 Tank	650.7	645.6	5.15^{13}	5.08^{13}	1.263	1.271
	144	S-106	640.1	638.7	5.02^{13}	5.03^{13}	1.275	1.270
	142	L-6 Tank	652.9	646.0	5.05^{13}	5.04^{13}	1.293	1.282
	145	A-37	649.6	647.6	5.01^{13}	5.10^{13}	1.297	1.270
	143	L-6 Tank	652.7	648.8	5.06^{13}	5.04^{13}	1.290	1.287
	146	A-28	646.0	646.0	5.06^{13}	5.04^{13}	1.277	1.282
			$\bar{X}$ = 548.59		$\bar{X}$ = 5.049 x 10^{13}		$\bar{X}$ = 1.2847	
R-2	189	L-6 Tank	780.8	788.0	5.94^{13}	6.08^{13}	1.314	1.296
	222	A-41	772.2 RR					1.287
			762.9	778.7	6.02^{13}	6.04^{13}	1.267	1.289
	220	L-6 Tank	780.5	784.6	6.09^{13}	6.06^{13}	1.282	1.295
	223	A-25	778.2	786.0	6.07^{13}	6.13^{13}	1.282	1.282
	221	L-6 Tank	781.7	--	5.86^{13}	6.07^{13}	1.334	1.288
	224	S-31	784.7	786.3	5.99^{13}	6.08^{13}	1.310	1.293
			$\bar{X}$ = 780.80		$\bar{X}$ = 6.036 x 10^{13}		$\bar{X}$ = 1.2938	
R-3	287	L-6	829.9	830.8	6.37^{13}	6.25^{13}	1.303	1.329
	311	L-6 Tank	830.0	830.8	5.94^{13}	6.23^{13}	1.397	1.333
					6.29^{13} RR	6.10^{13}	1.320	1.362
	314	A-20	331.2	836.0	6.35^{13}	6.22^{13}	1.309	1.344
	312	L-6 Tank	327.0	838.0	6.18^{13}	6.26^{13}	1.338	1.339
	315	A-24	839.6	827.6	6.17^{13}	6.11^{13}	1.361	1.355
	313	L-6 Tank	332.4	833.2	6.30^{13}	6.27^{13}	1.321	1.329
	316	A-12	328.9	827.8	6.21^{13}	6.16^{13}	1.335	1.344
			$\bar{X}$ = 831.66		$\bar{X}$ = 6.213 x 10^{13}		$\bar{X}$ = 1.3387	
R-4	337	L-6 Tank	359.4	863.7	6.30^{13}	6.40^{13}	1.364	1.350
	345	L-6 Tank	358.2	858.4	6.32^{13}	6.36^{13}	1.358	1.350
	348	S-136	348.4	868.8	6.28^{13}	6.30^{13}	1.351	1.379
	346	L-6 Tank	357.0	857.6	6.36^{13}	6.39^{13}	1.347	1.342
	349	S-134	845.0	852.4	6.37^{13}	6.36^{13}	1.327	1.340
	347	L-6 Tank	852.9	855.2	6.40^{13}	6.34^{13}	1.333	1.349
	350	S-129	844.4	878.4	6.28^{13}	6.40^{13}	1.345	1.373
			$\bar{X}$= 857.13		$\bar{X}$ = 6.347 x 10^{13}		$\bar{X}$ = 1.3506	

TABLE XII (Cont.)

L-6 PLUTONIUM ANALYTICAL VALUES

Run No.	Sample No.	Sample Type	Method: PUX-3A grams/gallon Original	PUX-3A Referee	PUA-6B-1 counts/min/gallon Original	PUA-6B-1 Referee	F-Factor grams/count min. Original	F-Factor Referee
R-5	366	L-6 Tank	540.3	--	4.04^{13}	3.96^{13}	1.337	--
	388	L-6 Tank	--	--	4.08^{13}	4.07^{13}	--	--
	402	L-6 Tank	562.6	560.6	4.14^{13}	4.26^{13}	1.359	1.316
	403	A-14	573.3	570.8	4.27^{13}	4.22^{13}	1.343	1.353
	404	L-6 Tank	--	--	4.15^{13}	4.13^{13}	--	--
	405	A-19	552.4	556.4	4.17^{13}	4.11^{13}	1.325	1.354
			$\bar{X}$ = 559.49		$\bar{X} = 4.133 \times 10^{13}$		$\bar{X}$ = 1.3410	
			$\bar{\bar{X}}$ = 754.3				$\bar{\bar{X}}$ = 1.321	
		(PuX-3a)	$s^{(6)}$ = 0.95%				S = 1.36%	

Run No.	Sample No.	Can No.	Method: PUA-6B-1 c/m/gallon Original	PUA-6B-1 Referee
R-6(7)	415	L-6 Tank	1.33^{13}	1.34^{13}
	416	L-6 Tank	1.32^{13}	1.35^{13}
	417	S-122	1.45^{13}	1.47^{13}
	418	S-127	1.38^{13}	1.37^{13}
				1.62^{13} RR
				1.65^{13} RR
			$\bar{X} = 1.428 \times 10^{13}$	
R-7	466	L-6 Tank	5.98^{12}*	3.00^{12}
				3.02^{12}
	477	A-45	3.04^{12}	3.19^{12}
	476	L-6 Tank	2.98^{12}	3.02^{12}
	482	A-47	3.30^{12}	3.37^{12}
	483	L-6 Tank	2.87^{12}	--
	484	S-124	2.88^{12}	3.09^{12}
				3.10^{12} RR
	485	L-6 Tank	3.04^{12}	3.10^{12}
	486	S-101	3.09^{12}	3.10^{12}
			$\bar{X} = 3.074 \times 10^{12}$	
R-8	495	L-6	1.31^{12}	1.33^{12}
	496	A-24	1.37^{12}	1.34^{12}
	512	L-6 Tank	1.28^{12}	1.28^{12}
	513	A-6	1.35^{12}	1.32^{12}
	516	L-6 Tank	1.28^{12}	1.30^{12}
	530	S-123	1.37^{12}	1.40^{12}
	531	A-20	1.48^{12}	1.52^{12}
			$\bar{X} = 1.353 \times 10^{12}$	

* Flier thrown Out

TABLE XII (Cont.)

Run No.	Sample No.	Can No.	Method: PUA-6B-1, c/m/gallon — Original	Referee
R-9	529	S-138	1.97^{12}	--
	542	A-28	2.00^{12}	$1.[illegible]^{12}$
			$\bar{X} = 1.987 \times 10^{12}$	
R-10	556	L-4	1.31^{12}	1.32^{12}
R-11	-	L-4 Tank	1.11^{12}	1.11^{12}
			$\bar{X} = 4.2[illegible] \times 10^{13}$	S = 2.10%

(1) Raw data uncorrected for analytical biases.

(2) One sample of L-6 tank was taken with each PR Can load-out with 15 minutes of circulation prior to each sample. Can numbers, e.g., S-106, represent thief samples of the respective cans.

(3) Plutonium by X-ray Absorption.

(4) Plutonium by direct Radioassay.

(5) "F" = grams/gallon/c/m/gallon = grams/c/m

(6) The standard deviation of an individual result on the same run - obtained from the pooled variance and includes sampling and analytical variance but not between run variance.

(7) Runs 6, 7, 8, 9, 10, 11 measured by Radioassay using an "F" factor based on R-4 and R-5 of 1.347×10^{-11} grams Pu/count/minute.

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TABLE XIII

INCIDENTAL ANALYSES

Type Sample	Det'n	Method	Number of Analyses	$\bar{\bar{X}}$	% s [(1)]
H-7	SpG	DZ-1A	74	1.6856	0.21
H-7	AMCM	AMCM-1B	73	1.67×10^9	9.56
H-7	HNO_3	HPH-1B-3	74	0.190	23.87
E-12	SpG	DW-1A	68	1.6512	0.08
E-12	SpG	DZ-1A	32	1.655	0.08
E-12	HNO_3	HPH-1B	32	-0.051	5.5
L-6	SpG	DZ-1A	69	1.4546	0.32
L-6	FE	FES-1F	66	0.1043	4.66
L-6	HNO_3	HV-3E	68	2.2466	5.38

(1) The standard deviation of an individual result on the same batch of material - obtained from the pooled variance and includes sampling and analytical variance but not between batch variance.

[redacted]

TABLE XIV

D-9 PLUTONIUM AND URANIUM ANALYTICAL VALUES[1]

Batch	Sample[4]	Uranium-Pounds/gallon U by UF-1b[2] Original	Uranium-Pounds/gallon U by UF-1b[2] Referee	Plutonium gram/gallon PU by PUA-20a Original	Plutonium gram/gallon PU by PUA-20a Referee
PW-2 RES	1	5.9×10^{-3}	4.5×10^{-3}	2.94×10^{-3}	2.20×10^{-3}
	2	5.3×10^{-3}	4.5×10^{-3}	3.74×10^{-3}	2.90×10^{-3}
		$\bar{X} = 5.1 \times 10^{-3}$		$\bar{X} = 2.95 \times 10^{-3}$	
PW-3	1	1.0×10^{-2}	6.4×10^{-3}	2.69×10^{-3}	2.63×10^{-3}
	2	9.8×10^{-3}	6.3×10^{-3}	2.05×10^{-3}	2.10×10^{-3}
		$\bar{X} = 8.1 \times 10^{-3}$		$\bar{X} = 2.37 \times 10^{-3}$	
PW-4	1	9.7×10^{-3}	2.1×10^{-2}	7.37×10^{-3}	4.81×10^{-3}
	2	8.4×10^{-3}	1.8×10^{-2}	6.33×10^{-3}	5.88×10^{-3}
			RR[5] 2.1×10^{-2}	$\bar{X} = 6.10 \times 10^{-3}$	
		$\bar{X} = 1.6 \times 10^{-2}$			
PW-5 + H-3	1	2.6×10^{-2}	2.2×10^{-2}	5.03×10^{-3}	8.37×10^{-3}
	2	2.4×10^{-2}	2.3×10^{-2}	4.07×10^{-3}	9.97×10^{-3}
		$\bar{X} = 2.4 \times 10^{-2}$			1.23×10^{-2} RR
					9.59×10^{-3} RR
					1.20×10^{-2} RR
				$\bar{X} = 9.07 \times 10^{-3}$	
PW-6	1	1.3×10^{-2}	1.1×10^{-2}	3.66×10^{-3}	4.75×10^{-3}
		1.2×10^{-2}	1.2×10^{-2}	3.60×10^{-3}	4.59×10^{-3}
		$\bar{X} = 1.2 \times 10^{-2}$		$\bar{X} = 4.15 \times 10^{-3}$	
PW-7	1	1.0×10^{-2}	1.5×10^{-2}	4.18×10^{-3}	3.79×10^{-3}
	2	1.2×10^{-2}	1.6×10^{-2}	3.88×10^{-3}	4.01×10^{-3}
		$\bar{X} = 1.3 \times 10^{-2}$		$\bar{X} = 3.97 \times 10^{-3}$	
PW-8	1	1.0×10^{-2}	1.2×10^{-2}	4.07×10^{-3}	3.76×10^{-3}
	2	1.5×10^{-2}	1.2×10^{-2}	4.11×10^{-3}	4.13×10^{-3}
		$\bar{X} = 1.2 \times 10^{-2}$		$\bar{X} = 4.12 \times 10^{-3}$	
PW-9	1	5.6×10^{-3}	6.2×10^{-3}	2.11×10^{-3}	1.46×10^{-3}
	2	4.5×10^{-3}	6.5×10^{-3} RR	2.31×10^{-3}	2.73×10^{-3}
			7.0×10^{-3}	$\bar{X} = 2.15 \times 10^{-3}$	
		$\bar{X} = 6.0 \times 10^{-3}$			
PW-11	1	9.6×10^{-3}	1.2×10^{-2}	5.46×10^{-3}	5.00×10^{-3}
	2	1.1×10^{-2}	1.3×10^{-2}	4.96×10^{-3}	5.19×10^{-3}
		$\bar{X} = 1.1 \times 10^{-2}$		$\bar{X} = 5.15 \times 10^{-3}$	

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TABLE XIV (Cont.)

D-9 PLUTONIUM AND URANIUM ANALYTICAL VALUES

		Uranium-Pounds gallon U by UF-[illegible]		Plutonium gram gallon PU by PUA-[illegible]	
Batch	Sample	Original	Referee	Original	Referee
PW-12	1	5.5×10^{-3}	5.2×10^{-3}	3.82×10^{-3}	6.40×10^{-3}
	2	5.6×10^{-3}	[illegible]$.6 \times 10^{-3}$	3.50×10^{-3}	6.30×10^{-3}
		$\bar{X} = 6.0 \times 10^{-3}$			5.06×10^{-3} RR
					4.20×10^{-3} RR
				$\bar{X} = 4.89 \times 10^{-3}$	
PW-13	1	2.1×10^{-3}	2.1×10^{-3}	2.80×10^{-3}	2.25×10^{-3}
	2	1.5×10^{-3}	2.1×10^{-3}	2.99×10^{-3}	2.31×10^{-3}
		$\bar{X} = 2.0 \times 10^{-3}$		$\bar{X} = 2.59 \times 10^{-3}$	
PW-14	1	1.5×10^{-2}	8.9×10^{-2}	1.67×10^{-3}	1.65×10^{-3}
	2	1.5×10^{-2}	1.1×10^{-3}	1.97×10^{-3}	1.74×10^{-3}
			9.9×10^{-4}	$\bar{X} = 1.76 \times 10^{-3}$	
			9.5×10^{-5}		
		$\bar{X} = 5.7 \times 10^{-3}$			
PW-15	1	1.4×10^{-3}	1.4×10^{-3}	2.58×10^{-3}	2.61×10^{-3}
	2	9.7×10^{-3}	1.3×10^{-3}	2.81×10^{-3}	2.72×10^{-3}
		$\bar{X} = 3.4 \times 10^{-3}$		$\bar{X} - 2.88 \times 10^{-3}$	
PW-16 & H-3	1	1.32×10^{-2}	4.4×10^{-3}	2.78×10^{-3}	5.70×10^{-3}
	2	4.7×10^{-3}	4.0×10^{-3}	5.36×10^{-3}	4.80×10^{-3}
		5.1×10^{-3} RR	4.7×10^{-3} RR	$\bar{X} = 4.66 \times 10^{-3}$	
		$\bar{X} = 6.0 \times 10^{-3}$			
PW-17	1	1.7×10^{-2}	1.4×10^{-2}	5.16×10^{-3}	5.29×10^{-3}
	2	1.5×10^{-2}	1.3×10^{-2}	5.11×10^{-3}	5.16×10^{-3}
		$\bar{X} = 1.5 \times 10^{-2}$		$\bar{X} = 5.18 \times 10^{-3}$	
		$\bar{\bar{X}} = 9.1 \times 10^{-3}$		$\bar{\bar{X}} = 4.43 \times 10^{-3}$	
		$S^{(6)} = 38\%$		$S = 30\%$	

(1) Raw data uncorrected for analytical biases.

(2) Uranium by the conventional Hexone Extraction-Fluorimetric Uranium method.

(3) Plutonium by the conventional TTA Extraction-Total Alpha Counting Method using an "F" Factor of 1.271×10^{-11} grams Pu/count/min.

(4) Samples taken 15 minutes apart with full sampler circulation between samples and continuous tank agitation.

(5) Analytical Rerun.

(6) The standard deviation of an individual determination on the same batch - obtained from the pooled variance and includes sampling and analytical variance, but not between-batch variance.

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TABLE XV

H-10 PLUTONIUM AND URANIUM ANALYTICAL VALUES (1)

Batch No.	Sample	Uranium-Pounds/Gallon, U by UF-1b (2): Original	Uranium-Pounds/Gallon, U by UF-1b (2): Referee	Plutonium - Grams/Gallon, PU by FUA-[illegible]a (3): Original	Plutonium - Grams/Gallon, PU by FUA-[illegible]a (3): Referee
D-4	1 (4)	1.5×10^{-2}	1.4×10^{-2}	2.61×10^{-3}	$2.41 \times 10^{-[illegible]}$
	2	1.5×10^{-2}	1.6×10^{-2}	2.42×10^{-3}	$2.19 \times 10^{-[illegible]}$
		$\bar{X} = 1.5 \times 10^{-2}$		$\bar{X} = 2.4 \times 10^{-3}$	
D-2	1	1.1×10^{-2}	7.0×10^{-3}	2.25×10^{-3}	1.70×10^{-3}
	2	2.2×10^{-2}	2.0×10^{-2}	2.06×10^{-3}	2.49×10^{-3}
		$\bar{X} = 1.5 \times 10^{-2}$		$\bar{X} = 2.13 \times 10^{-3}$	
#20	1	3.6×10^{-3}	3.1×10^{-3}	7.78×10^{-4}	8.[illegible] $\times 10^{-4}$
	2	3.6×10^{-3}	4.0×10^{-3}	1.14×10^{-3}	9.52×10^{-4}
		$\bar{X} = 3.6 \times 10^{-3}$		$\bar{X} = 9.27 \times 10^{-4}$	
#19	1	2.7×10^{-3}	2.5×10^{-3}	7.8×10^{-4}	7.4×10^{-4}
	2	2.6×10^{-3}	2.5×10^{-3}	7.8×10^{-4}	6.5×10^{-4}
		$\bar{X} = 2.6 \times 10^{-3}$		$\bar{X} = 7.37 \times 10^{-4}$	
#18	1	5.5×10^{-4}	9.0×10^{-4}	5.7×10^{-4}	4.6×10^{-4}
	2	1.1×10^{-3}	1.4×10^{-3}	6.1×10^{-4}	6.2×10^{-4}
			1.4×10^{-3}	$\bar{X} = 5.65 \times 10^{-4}$	
			$\bar{X} = 1.1 \times 10^{-3}$		
C-1	1	0.506	0.505	5.2×10^{-2}	4.6×10^{-2}
	2	0.584	0.505	6.1×10^{-2}	5.9×10^{-2}
	3	0.745		6.6×10^{-2}	
	4	0.747		6.4×10^{-2}	
		$\bar{X} = 0.599$		$\bar{X} = 0.058$	
		$\bar{\bar{X}} = 0.140$		$\bar{\bar{X}} = 1.4 \times 10^{-2}$	
		$s^{(5)} = 20\%$		$s^{(5)} = 12\%$	

(1) Raw data uncorrected for analytical biases.

(2) Uranium by the conventional Hexone Extraction-Fluorimetric Uranium method.

(3) Plutonium by the conventional TTA Extraction-Total Alpha Counting method using an "F" factor of 1.274×10^{-11} grams Pu/count/minute.

(4) Samples taken 15 minutes apart with full sampler circulation between samples and continuous tank agitation.

(5) The standard deviation of an individual result on the same batch - obtained from the arithmetic average of batch % standard deviation per individual result and includes sampling and analytical variance but not between batch variance

G. Bias Control

In conjunction with the analytical phase of the test program, an extensive analytical control standard analysis program was carried out for the purpose of bias control. In all cases, the control standards were analyzed in a manner identical to that used for the process samples. In addition, the composition of standards, insofar as it was practical, approximated the composition of the process samples. The number of control standards analyzed by the various analytical methods employed and the recoveries and precisions obtained are illustrated in Table XVI. The various analytical methods referred to in code in Table XVI are more fully described in Table XVII.

From the control standard recoveries shown in Table XVIII, analytical bias corrections were applied to H-7, E-12, and L-6 totals to obtain the corrected material balance previously discussed. Because of the small amount of material involved, no bias corrections were applied to D-9 and E-10 values. The magnitude and direction of the bias corrections used are given in Table XVIII. In addition to the conventional analytical bias correction (that based on control standard recoveries) applied to L-6 X-ray results, a correction of 0.2% was applied to the plutonium X-ray results to compensate for the amount of chromium and nickel (determined by spectrographic analysis) present in the process samples but not corrected for in normal control laboratory practice.

The most striking example of the worth of analytical bias corrections occurring in the test run is illustrated by the E-12 uranium results. Two methods, Uranium by X-ray (UX-2a) and Uranium by Density (UD-1a), which under unbiased conditions produce identical results were used for all E-12 process samples. As the raw data (Shown in Table XI) shows, the UD-1a values are consistently higher than (higher on the average by 0.71%) than the X-ray values, indicating the existence of a definite bias. This is further borne out by the control standard recovery values of 100.7% for UD-1a and 100.0% for UX-2a. After making the necessary analytical bias correction to the UD-1a values. the two corrected values for the process samples now agree within 0.01%.

H. "F" Factor Determination

For the sake of simplicity, a constant "F" factor of 1.271×10^{-11} gram Pu/alpha count/minute was used by the Control Laboratory throughout the test run to convert H-7 alpha total values to plutonium weight units. After completion of the test run, a weighted (weighted in terms of plutonium produced) "F" factor of 1.321×10^{-11} was computed from the "F" factors obtained at L-6. This "F" factor which represents the uncorrected "F" factor for the entire test run batch was used to adjust H-7 receipts to the values previously discussed. From the uncorrected "F" factor a corrected "F" factor of 1.299×10^{-11} was obtained by the application of the analytical bias corrections previously described.

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TABLE XVI

QUALITY CONTROL STANDARD RECOVERIES

Std.	Method	Det'n	#Anal.	Avg. % Recovery	Std. Dev. of Anal.	Comment
E-12	DZ-1a #4	SpG	13	100.6	0.50%	
H-7	DZ-1a #5	SpG	17	100.4	0.24%	
E-12	UD-1a	U	16	100.7	0.38%	By DW-1a
H-7	UD-1a	U	17	101.2	0.52%	By DZ-1a
E-12	DW-1a	SpG	16	100.1	0.07%	
E-12	HPH-1b #3	HNO_3	17	91.3	14.52%	
H-7	HPH-1b #3	HNO_3	17	105.5	6.97%	
E-12	UX-2a	U	19	100.0	0.18%	
H-7	UX-4a	U	24	100.5	0.88%	
ANCN	ANCN-1b	ANCN	20	117.9	21.44% (for H-7)	
H-7	PuA-6b	AT	27	99.6	1.22%	
D-9	PuA-20a	Pu	32	95.9	9.72%	
D-9	UF-1b	U	35	100.3	22.41%	
L-6	PUX-3a	Pu	44	100.3	0.80%	
L-6	PUA-6b	AT	64	98.8	0.44%	
L-6	F-Factor		41	101.5	1.16%	
L-6	FES-1F	FE	56	101.8	1.70%	
L-6	HV-3E	HNO_3	59	100.8	5.10%	
L-6	DZ-1a Tube #3	SpG	26	100.0	0.10%	

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TABLE XVII

DESCRIPTION OF ANALYTICAL METHOD CODES

Method Code	Determination	Descriptive Title
AmCm-1b	Americium-curium	TBP, TTA Separation - Total Alpha Counting
DW-1a	Density or specific gravity	Density by Pycnometer.
DZ-1a	Density or specific gravity	Density by Falling Drop
"F" Factor	Grams Pu/alpha count/minute	Plutonium Specific Alpha Activity Factor.
FeS-1f	Iron	Colorimetric-Orthophenathroline.
HpH-1b	Nitric Acid	Nitric acid by pH.
HV-9e	Nitric acid	Nitric acid-oxalate complexing of plutonium-microtitration.
PUA-6b	Alpha total	Direct Radioassay.
PUA-20a	Plutonium alpha	TTA Separation - Total Alpha Counting
PUX-3a	Plutonium	Direct X-ray Absorption.
UF-1b	Uranium	Hexone Extraction - Uranium Fluorescence.
UX-2a	Uranium	Direct X-ray Absorption.
UX-4a	Uranium	TBP Extraction-X-ray Absorption.
UD-1a	Uranium	Uranium calculated from Density.

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TABLE XVIII

ANALYTICAL BIAS CORRECTIONS APPLIED TO MEASUREMENT VALUES

Measure-ment Point	Determination	Method Code	% Control Std. Recovery	Measure-ment Value changed	% Measure-ment Value changed
H-7	specific gravity(1)	DZ-1a	100.4	Volume	+ 0.4
H-7	Americium-curium	AmCm-1b	117.9	plutonium alpha	+ 0.3
H-7	uranium	UX-4a	100.5	uranium	- 0.5
H-7	uranium	UD-1a	101.2	uranium	- 1.2
H-7	alpha total (Pu)	PuA-6b	99.6 (2)	alpha total	+ 0.4
H-7	plutonium	"F" Factor	101.5(+0.2)	plutonium	- 1.5
E-12	uranium	UX-2a	100.0	N. C.(3)	0.0
E-12	uranium	UD-1a	100.7	uranium	- 0.7
E-12	specific gravity	DW-1a	100.1	volume	+ 0.1
L-6	plutonium	PUX-3a	100.3(+0.2)	plutonium	+ 0.5
L-6	specific gravity	DZ-1a	100.0	N.C.	0.0

(1) May be interchanged with density.

(2) Correction for chromium and nickel present in process L-6 samples not compensated for by present method. Chromium and nickel values determined by spectrographic analysis.

(3) No change in measurement value.

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