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16. KEY

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2	1	Cog.Eng. G.L. Miller per telecon 1/25/96			
2	1	Cog. Mgr. J.L. Deichman 1/23/96			
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		Safety			
		Env.			
2	1	Production Planning and Control - A.D. Rice A.D. Rice 1-22-96			
2	1	C.L. Bennett C.L. Bennett 1/24/96			

18. L. R. Webb per telecon 1/25/96
Signature of EDT Originator — Date

19. C. Defigh-Price 1/24/96
Authorized Representative for Receiving Organization — Date

20. J.L. Deichman 1/23/96
Cognizant Manager — Date

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105-K East Sandfilter Backwash Line Sample Analysis Report - Third Campaign

George L. Miller
Westinghouse Hanford Company, Richland, WA 99352
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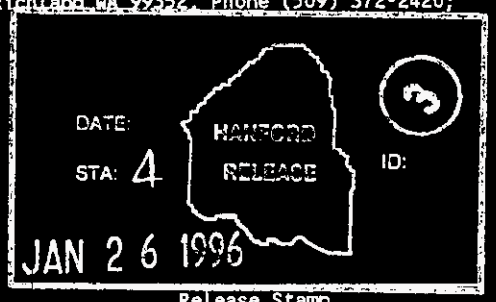

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ANALYTICAL SERVICES

105-K EAST SANDFILTER BACKWASH LINE SAMPLE ANALYSIS REPORT - THIRD CAMPAIGN

Project Coordinator: GEORGE L. MILLER

Prepared for the U.S. Department of Energy
Office of Environmental Restoration
and Waste Management

by

Westinghouse Hanford Company
Box 1970

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This document consists of pages 1 through 27.

105-K EAST SANDFILTER BACKWASH LINE SAMPLE ANALYSIS REPORT THIRD CAMPAIGN

D. B. Bechtold

1.0 INTRODUCTION

This project seeks to produce uranium (U) and plutonium (Pu) analyses of samples taken from the KE basin filter backwash line each time the sand filter is backwashed. K Basin operations will use the analytical results to determine additions of fissile materials to the backwash sludge pit and thereby maintain a running inventory of fissile elements in the pit. K Basin operations must not exceed a certain total inventory in order to be within a criticality specification.

The third campaign of this project consisted of three samples, numbered by the customer 245KEB, 246KEB, and 247KEB. A revised letter of instruction controlled their processing (Reference 4.1).

2.0 SAMPLE PREPARATION

All work on the samples except the actual analysis of digests is recorded in controlled laboratory notebook WHC-N-341-2, pages 129-134.

2.1 Appearance and Dose Rates.

All three samples arrived in 250 mL jars virtually full of clear water, with a very small amount of brown, flocculent sludge lying on the bottom -- not enough to cover the bottom. Over-the-Top Reading (OTR) dose rates are included in the Sample Preparation Data Sheet.

2.2 Sludge Settling.

Because so little sludge was present in each sample, no attempts at sludge volume or mass determinations were made. Each jar was merely marked at the total sample level, then allowed to settle for 24 hours before proceeding. The sludge settled well, leaving clear, excess water.

2.3 Sample Preparation.

Due to complete settling, the excess water was pipetted off each sample before the sludge contents of each sample were transferred individually to PTFE drying/digestion beakers. No excess water was submitted for total alpha analysis, since this was not requested on the chain of custody. The cleaned sample jars were

tared, then weighed with laboratory water to the total sample (water) level marks.

Each sample was dried in a PTFE beaker with a PTFE watchglass lid and PTFE standoffs on a hot plate to yield in all cases dark brown material like broken up dried mud. All samples yielded less than 0.3 gram each of dried sludge; therefore, all sludges were digested and no excess sludge was archived.

Each sample was digested twice with approximately 50 mL of acid that was approximately 5M each in nitric and hydrochloric acids, and 3 drops concentrated HF, for four hours. Digestates were separated from residues by decantation/rinsing/centrifugation and placed into 500 mL volumetric flasks for dilution to known volume. Samples from each diluted digestate were submitted for total U analysis, Pu^{238} analysis and $Pu^{239,240}$ analysis. The remaining digestates were archived.

2.4 Residues

The undigested residues for each sample were dried to constant weight in the digesters, weighed and observed. There was very little residue in any sample (less than 200 mg in all cases). The residues appeared to consist of tiny black bits.

3.0 RESULTS

3.1 Sample Preparation Results

The sample preparation datasheet appears as Table 1, where the procedural steps are described in Reference 4.1. All data precision and accuracy requirements for sample preparation listed in the letter of instruction (LOI) (Reference 4.1) were judged to have been met, except for steps to measure sludge weight and volume, which were not performed due to insufficient sludge.

TABLE 1. SAMPLE PREPARATION DATA, THIRD CAMPAIGN

STEP	PARAMETER DESCRIPTION	UNITS	SAMPLE RESULTS		
1	Sample identity		CUSTOMER NO. 245KEB	CUSTOMER NO. 246KEB	CUSTOMER NO. 247KEB
	amount of sludge	mL	3	3	3
	contact dose rate	mRad or mRem	2.5	2.5	2.0
3	Gross weight of sample bottle	g	413.302	436.512	428.029
9	Net weight of excess water (if requested)	g	n/a	n/a	n/a
	Net weight of laboratory water sample	g	n/a	n/a	n/a
	water sample number		n/a	n/a	n/a
10	Gross wt. of sample bottle w/o excess water	g	n/a	n/a	n/a
11	Tare weight of PTFE drying/digesting vessel	g	139.842	142.377	140.544
15	Tare weight of sample bottle	g	214.948	215.377	218.585
17	Weight of bottle filled to sludge mark	g	n/a	n/a	n/a
19	Wt. of bottle filled to initial liquid level	g	416.757	440.588	433.479
20	Gross wt. of drying vessel, first drying	g	139.904	142.605	140.685
	second drying	g	139.904	142.604	140.686
	third drying	g	n/a	n/a	n/a
24	Net weight of excess dry solids	g	0	0	0
25	Gross weight solids to be digested	g	139.904	142.604	140.686
40	Volume of diluted digestate	mL	500	500	500
44	Gross weight of dry digester plus residue	g	139.87	142.565	140.653
	second digestion drying	g	139.866	142.561	140.65
	third digestion drying	g	n/a	n/a	n/a
46	Appearance of dried residue		Some minute black bits	Some minute black bits	Some minute black bits

3.2 Radiochemical Results

Table 2 includes the analytical laboratory radiochemical results for this, the third campaign.

Table 2. Radiochemical Results, Third Campaign

DATUM	UNITS	CUSTOMER # 245KEB	CUSTOMER # 246KEB	CUSTOMER # 247KEB
PCS# DIGEST SAMPLE	none	JMK133A	JMK133B	JMK133C
LAB# DIGEST SAMPLE	none	S95R000001	S95R000002	S95R000003
LAB1 U	ug/mL digest	1.09e+01	1.22e+01	1.16e+01
LAB2 U	ug/mL digest	1.05e+01	1.22e+01	1.13e+01
LAB3 U	ug/mL digest	1.05e+01	1.21e+01	1.12e+01
LAB U DET. LIM.	ug/mL digest	3.70e-04	3.70e-04	3.70e-04
LAB U BLANK	ug/mL	6.04e-05	6.04e-05	6.04e-05
U RSD	%	2.2	0.5	1.8
LAB U SPIKE RECOV.	%	91.4	n/a	n/a
LAB U STD. RECOV.	%	94.8	94.8	94.8
U DET. LIM.	ug/mL sample	9.15e-04	8.20e-04	8.59e-04
LAB1 Pu^{238}	uCi/mL digest	4.28e-04	<3.48e-04	4.57e-04
LAB2 Pu^{238}	uCi/mL digest	4.24e-04	<6.71e-04	<6.79e-04
LAB3 Pu^{238}	uCi/mL digest	4.33e-04	<4.28e-04	<9.43e-04
Pu^{238} RSD	%	1.1	n/a	n/a
LAB Pu^{238} COUNT ERROR	%	3.7	6.0	3.7
LAB1 $Pu^{239,240}$	µCi/mL digest	2.73e-03	2.65e-03	2.99e-03
LAB2 $Pu^{239,240}$	µCi/mL digest	2.73e-03	3.04e-03	3.15e-03
LAB3 $Pu^{239,240}$	µCi/mL digest	2.79e-03	2.98e-03	2.95e-03
LAB $Pu^{239,240}$ DET. LIM.	µCi/mL digest	2.40e-04	3.48e-04	2.73e-04
LAB $Pu^{239,240}$ BLANK	µCi/mL digest	<5.68e-05	<2.59e-04	<7.91e-05
$Pu^{239,240}$ RSD	%	1.3	7.3	3.5
LAB $Pu^{239,240}$ SPIKE RECOV.	%	96.7	98.2	112.6
LAB $Pu^{239,240}$ STD. RECOV.	%	100.0	89.8	96.9
LAB $Pu^{239,240}$ COUNT ERROR	%	2.3	3.0	2.4
$Pu^{239,240}$ DET. LIM.	µCi/mL sample	5.93e-04	7.71e-04	6.34e-04
U/$Pu^{239,240}$ RATIO	µg U/µCi $Pu^{239,240}$	3.87e+03	4.21e+03	3.75e+03

Examination of Table 2 reveals that all precision, accuracy and minimum detection limit requirements of the LOI Table 1 (Reference 4.1), when converted to comparable units, have been met for this campaign.

3.3 Auxiliary Results

Table 3 includes the complete third campaign results. It is presented as an aid to compare this campaign with previous ones.

Table 3. Complete Third Campaign Results

DATUM	UNITS	CUSTOMER # 245KEB	CUSTOMER # 246KEB	CUSTOMER # 247KEB
CAMPAIGN	none	THIRD	THIRD	THIRD
SLUDGE	mL	3	3	3
DOSE RATE	mRad or mRem	2.5	2.5	2
SLUDGE APPEARANCE	none	clear water,brown floc	clear water,brown floc	clear water,brown floc
GROSS SAMPLE JAR	g	413.302	436.512	428.029
GROSS NO WATER	g	n/a	n/a	n/a
NET EXCESS WATER	g	n/a	n/a	n/a
TARE WATER SAMPLE	g	n/a	n/a	n/a
GROSS WATER SAMPLE	g	n/a	n/a	n/a
NET WATER SAMPLE	g	n/a	n/a	n/a
PCS# WATER SAMPLE	none	n/a	n/a	n/a
LAB# WATER SAMPLE	none	n/a	n/a	n/a
TARE DIGESTER	g	139.842	142.377	140.544
TARE SAMPLE JAR	g	214.948	215.377	218.585
NET SLUDGE	g	n/a	n/a	n/a
GROSS SAMPLE JAR_TO_SLUDGE	g	n/a	n/a	n/a
GROSS SAMPLE JAR_TO_TOTAL	g	416.757	440.588	433.479
VOLUME SAMPLE	mL	2.02e+02	2.26e+02	2.15e+02
GROSS DRY 1ST	g	139.904	142.605	140.685
GROSS DRY 2ND	g	139.904	142.604	140.686
GROSS DRY 3RD	g	n/a	n/a	n/a
DRIES TO AVERAGE	none	1.2	2.	1.2
AVERAGE GROSS DRY	g	139.904	142.604	140.685
NET DRY SOLIDS	g	0.062	0.227	0.142
GROSS DIGEST	g	139.904	142.604	140.685
NET EXCESS SOLIDS	g	0.000	0.000	0.000
NET DIGEST SOLIDS	g	0.062	0.227	0.142
VOLUME DIGESTATE	mL	500	500	500
PCS# DIGEST SAMPLE	none	JMK133A	JMK133B	JMK133C
LAB# DIGEST SAMPLE	none	S95R000001	S95R000002	S95R000003
GROSS RESIDUE 1ST	g	139.870	142.565	140.653
GROSS RESIDUE 2ND	g	139.866	142.561	140.650
GROSS RESIDUE 3RD	g	n/a	n/a	n/a

Table 3. Complete Third Campaign Results

DATUM	UNITS	CUSTOMER # 245KEB	CUSTOMER # 246KEB	CUSTOMER # 247KEB
RESIDUES TO AVERAGE	none	2,	2,	2,
GROSS AVERAGE RESIDUE	g	139.866	142.561	140.650
NET RESIDUE	g	0.024	0.184	0.106
APPEARANCE RESIDUE	none	Some minute black bits	Some minute black bits	Some minute black bits
SLUDGE VOLUME	mL	n/a	n/a	n/a
SLUDGE DENSITY	g/mL sludge	n/a	n/a	n/a
WT % SOLIDS	% of sludge	n/a	n/a	n/a
WT % WATER	% of sludge	n/a	n/a	n/a
SOLIDS CONC	g/mL sludge	n/a	n/a	n/a
WATER CONC	g/mL sludge	n/a	n/a	n/a
AVG PARTICLE DENSITY	g/mL particle	n/a	n/a	n/a
WT % RESIDUE OF DIGEST	% of dry solids	3.87e+01	8.11e+01	7.49e+01
WT % RESIDUE OF SLUDGE	% of sludge	n/a	n/a	n/a
LAB1 U	µg/mL digest	1.09e+01	1.22e+01	1.16e+01
LAB2 U	µg/mL digest	1.05e+01	1.22e+01	1.13e+01
LAB3 U	µg/mL digest	1.05e+01	1.21e+01	1.12e+01
LAB U DET. LIM.	µg/mL digest	3.70e-04	3.70e-04	3.70e-04
LAB U BLANK	µg/mL	6.04e-05	6.04e-05	6.04e-05
U RSD	%	2.2	0.5	1.8
LAB U SPIKE RECOV.	%	91.4	n/a	n/a
LAB U STD. RECOV.	µg/mL	94.8	94.8	94.8
U DET. LIM.	µg/mL sample	9.15e-04	8.20e-04	8.59e-04
U SAMPLE 1	µg/mL sample	2.70e+01	2.70e+01	2.69e+01
U SAMPLE 2	µg/mL sample	2.60e+01	2.70e+01	2.62e+01
U SAMPLE 3	µg/mL sample	2.60e+01	2.68e+01	2.60e+01
U SAMPLE AVG	µg/mL sample	2.63e+01	2.70e+01	2.64e+01
U SOLIDS AVG	µg/g dry solids	8.58e+04	2.68e+04	4.02e+04
LAB1 Pu^{238}	µCi/mL digest	4.28e-04	<3.48e-04	4.57e-04
LAB2 Pu^{238}	µCi/mL digest	4.24e-04	<6.71e-04	<6.79e-04
LAB3 Pu^{238}	µCi/mL digest	4.33e-04	<4.28e-04	<9.43e-04
Pu^{238} RSD	%	1.1	n/a	n/a
LAB Pu^{238} COUNT ERROR	%	3.7	6.0	3.7
Pu^{238} SAMPLE 1	µCi/mL sample	1.06e-03	n/a	1.06e-03
Pu^{238} SAMPLE 2	µCi/mL sample	1.05e-03	n/a	n/a

Table 3. Complete Third Campaign Results

DATUM	UNITS	CUSTOMER # 245KEB	CUSTOMER # 246KEB	CUSTOMER # 247KEB
Pu^{238} SAMPLE 3	μCi/mL sample	1.07e-03	n/a	n/a
Pu^{238} SAMPLE AVG	μCi/mL sample	1.06e-03	n/a	1.06e-03
Pu^{238} SOLIDS AVG	μCi/g dry solids	3.45e+00	n/a	1.61e+00
LAB1 $Pu^{239,240}$	μCi/mL digest	2.73e-03	2.65e-03	2.99e-03
LAB2 $Pu^{239,240}$	μCi/mL digest	2.73e-03	3.04e-03	3.15e-03
LAB3 $Pu^{239,240}$	μCi/mL digest	2.79e-03	2.98e-03	2.95e-03
LAB $Pu^{239,240}$ DET. LIM.	μCi/mL digest	2.40e-04	3.48e-04	2.73e-04
LAB $Pu^{239,240}$ BLANK	μCi/mL digest	<5.68e-05	<2.59e-04	<7.91e-05
$Pu^{239,240}$ RSD	%	1.3	7.3	3.5
LAB $Pu^{239,240}$ SPIKE RECOV.	%	96.7	98.2	112.6
LAB $Pu^{239,240}$ STD. RECOV.	%	100.0	89.8	96.9
LAB $Pu^{239,240}$ COUNT ERROR	%	2.3	3.0	2.4
$Pu^{239,240}$ DET. LIM.	μCi/mL sample	5.93e-04	7.71e-04	6.34e-04
$Pu^{239,240}$ SAMPLE 1	μCi/mL sample	6.75e-03	5.87e-03	6.94e-03
$Pu^{239,240}$ SAMPLE 2	μCi/mL sample	6.75e-03	6.74e-03	7.31e-03
$Pu^{239,240}$ SAMPLE 3	μCi/mL sample	6.90e-03	6.60e-03	6.85e-03
$Pu^{239,240}$ SAMPLE AVG	μCi/mL sample	6.80e-03	6.40e-03	7.04e-03
$Pu^{239,240}$ SOLIDS AVG	μCi/g dry solids	2.22e+01	6.37e+00	1.07e+01
U/$Pu^{239,240}$ RATIO	μg U/μCi $Pu^{239,240}$	3.87e+03	4.21e+03	3.75e+03

One may compare the radiochemical data in Table 3 on a dry weight basis with the data from the backwash pit campaign (Reference 4.3), and the two previous backwash line campaigns (References 4.4 and 4.5), as shown in Figures 1 to 4. This campaign adds more information to these figures to suggest that the radiochemical content of backwash sludge is consistent with past, though highly variable, observations.

4.0 REFERENCES

4.1 Internal Memo, number 95-2A100.319, C. Defigh-Price to G. L. Miller, *Letter of Instruction for KE Basins Sandfilter Backwash Line Samples, Revision 1*, dated August 17, 1995.

4.2 WHC-SD-NR-TRP-023, REV 1, *Laboratory Test Plan for Analysis of KE Basin Backwash Pit Samples*, D. B. Bechtold, November 18, 1994, Westinghouse Hanford Company, Richland, Washington.

4.3 WHC-SD-NR-TRP-021, REV 0, *Report of Laboratory Test Plan for Analysis of KE Basin Backwash Pit Samples*, D. B. Bechtold, March 28, 1994, Westinghouse Hanford Company, Richland, Washington.

4.4 WHC-SD-SNF-DP-002, Rev. 0, *105-K East Sandfilter Backwash Line Sample Analysis Report, First Campaign*, D. B. Bechtold and G. L. Miller, November, 1995, Westinghouse Hanford Company, Richland, Washington.

4.5 WHC-SD-SNF-DP-005, Rev. 0, *105-K East Sandfilter Backwash Line Sample Analysis Report, Second Campaign*, D. B. Bechtold, B. A. Crawford and G. L. Miller, December, 1995, Westinghouse Hanford Company, Richland, Washington.

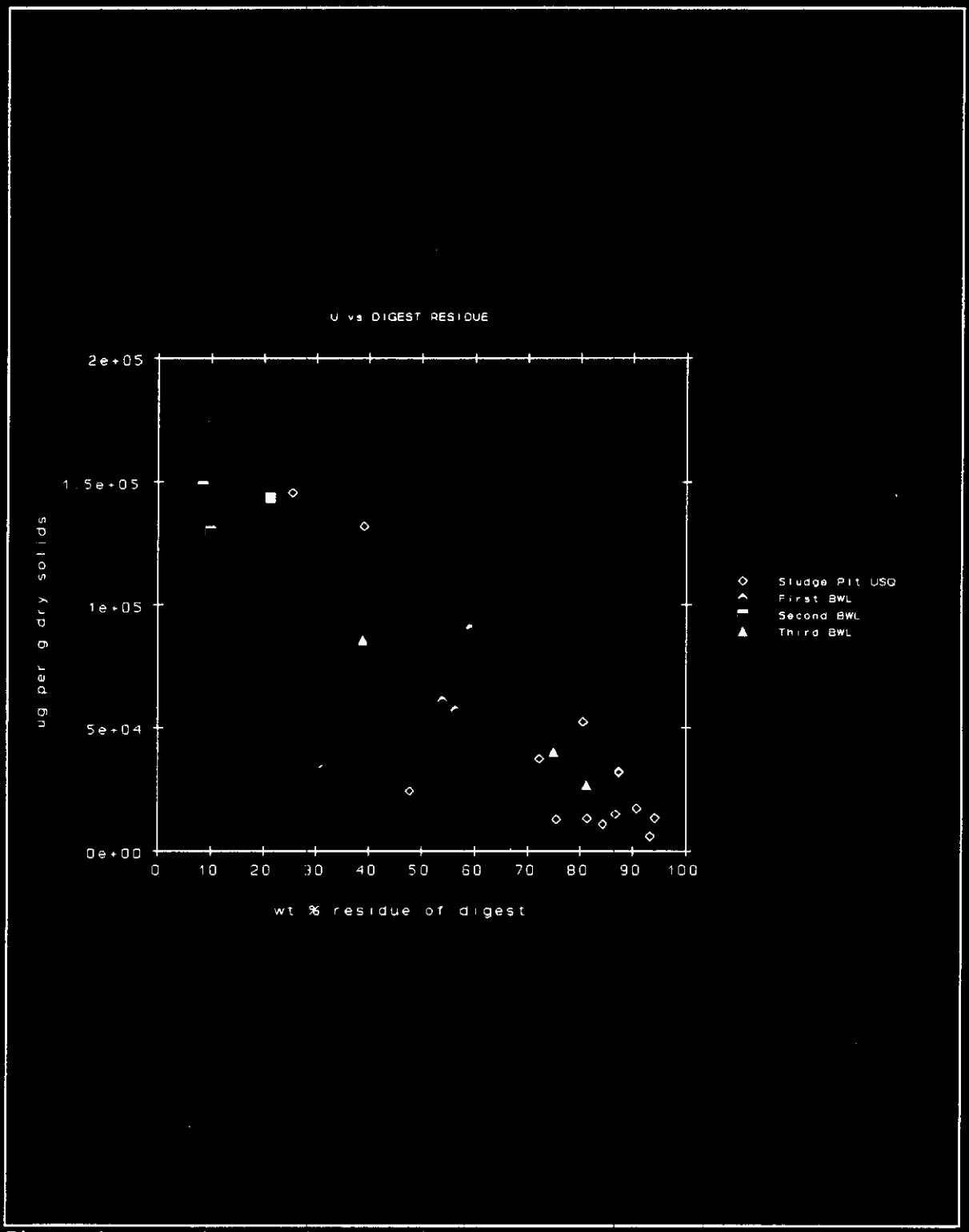

Figure 1. Uranium vs Digest Residue.

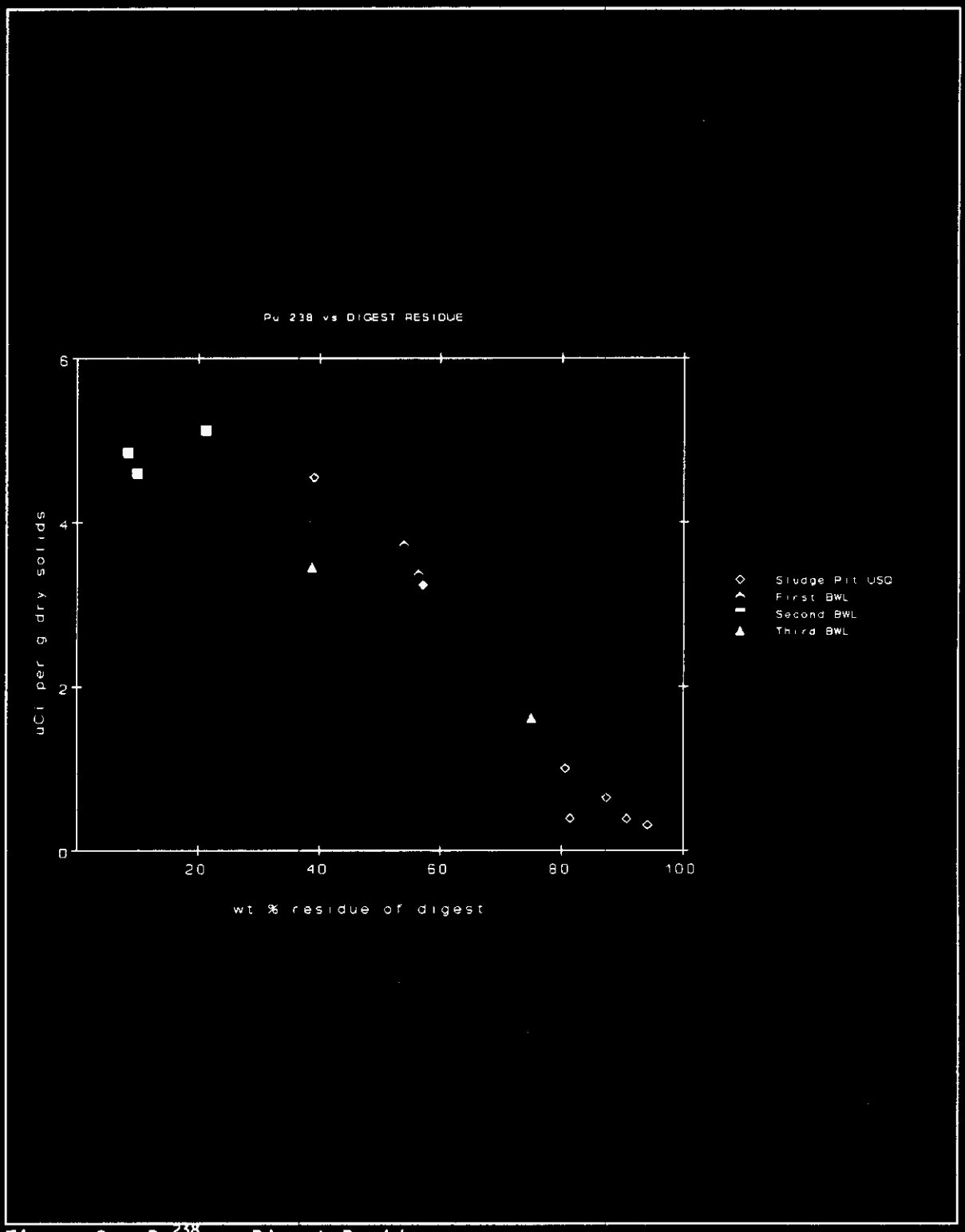

Figure 2. Pu^{238} vs Digest Residue.

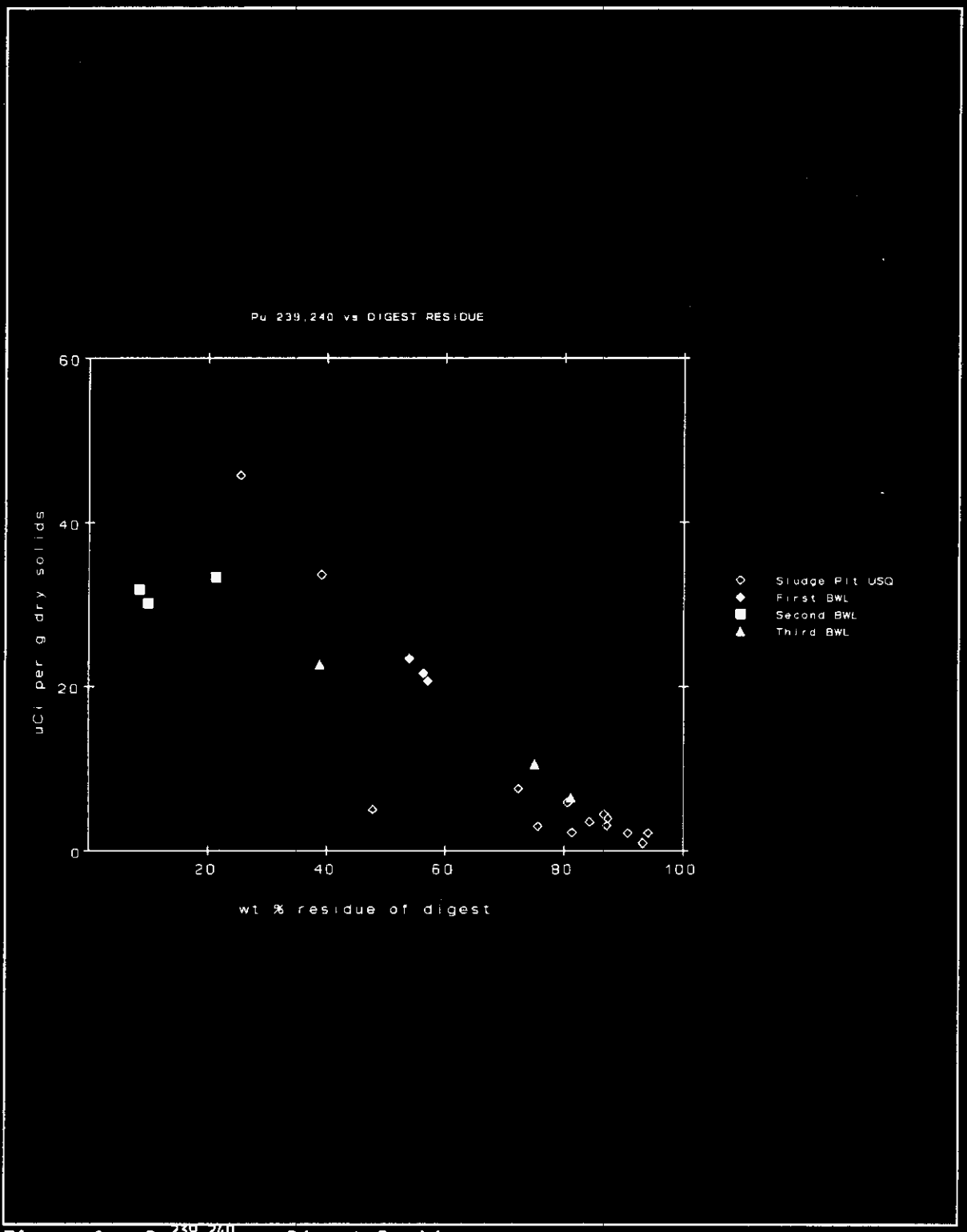

Figure 3. $Pu^{239,240}$ vs Digest Residue.

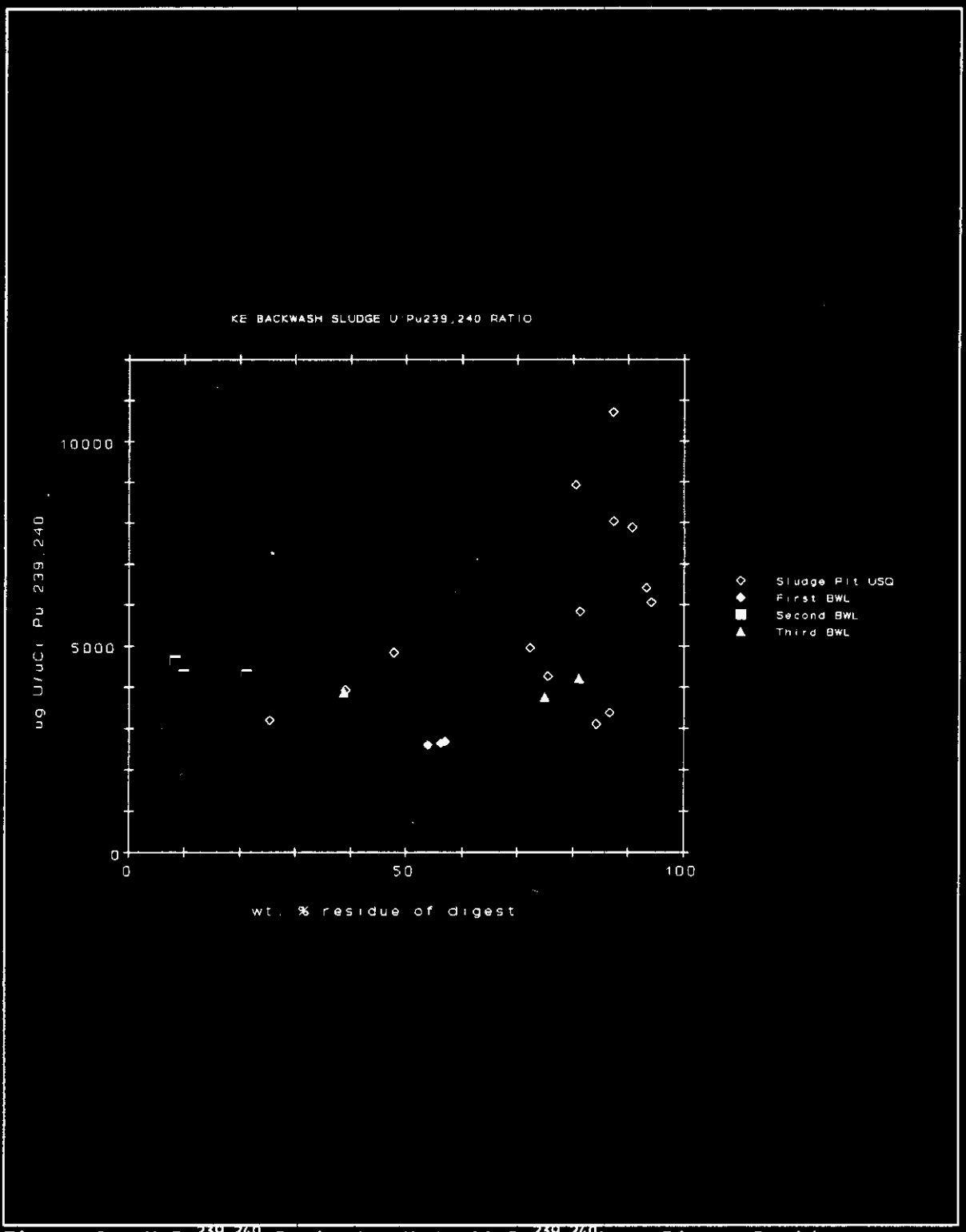

Figure 4. U:$Pu^{239,240}$ Ratio (μg U / μCi $Pu^{239,240}$) vs Digest Residue.

APPENDIX A

K-East Basin Sand Filter Backwash Line, Campaign III Activities/Concentrations of the Digested Samples

K-East Basin Sand Filter Backwash Line, Campaign III
Activities/Concentrations of the Digested Samples

Pu-239/240

SAMPLE IDENTIFICATION			SAMPLE DATA			QC DATA				
Sample #	PCS #	Customer #	Sample µCi/ml	Duplicate µCi/ml	Triplicate µCi/ml	Spike % Recovery	Blank µCi/ml	Standard % Recovery	Detection Limit µCi/ml	Rel. Counting Error %
S95R000001	JMK133A	245KEB	2.73E-03	2.73E-03	2.79E-03	96.7	<5.68E-05	100.0	2.40E-04	2.3
S95R000002	JMK133B	246KEB	2.65E-03	3.04E-03	2.98E-03	98.2	<2.59E-04	89.8	3.48E-04	3.0
S95R000003	JMK133C	247KEB	2.99E-03	3.15E-03	2.95E-03	112.6	<7.91E-05	96.9	2.73E-04	2.4

Pu-238

SAMPLE IDENTIFICATION			SAMPLE DATA			QC DATA			
Sample #	PCS #	Customer #	Sample µCi/ml	Duplicate µCi/ml	Triplicate µCi/ml	Blank µCi/ml	Pu-239 Standard % Recovery	Detection Limit µCi/ml	Rel. Counting Error %
S95R000001	JMK133A	245KEB	4.28E-04	4.24E-04	4.33E-04	<5.68E-05	100.0	2.40E-04	3.7
S95R000002	JMK133B	246KEB	<3.48E-04	<6.71E-04	<4.28E-04	<2.59E-04	89.8	3.48E-04	6.0
S95R000003	JMK133C	247KEB	4.57E-04	<6.79E-04	<9.43E-04	<7.91E-05	96.9	2.73E-04	3.7

Total Uranium

SAMPLE IDENTIFICATION			SAMPLE DATA			QC DATA			
Sample #	PCS #	Customer #	Sample µg/ml	Duplicate µg/ml	Triplicate µg/ml	Spike % Recovery	Blank µg/ml	Standard % Recovery	Detection Limit µg/ml
S95R000001	JMK133A	245KEB	1.09E+01	1.05E+01	1.05E+01	91.4	6.04E-05	94.8	3.70E-04
S95R000002	JMK133B	246KEB	1.22E+01	1.22E+01	1.21E+01	n/a	6.04E-05	94.8	3.70E-04
S95R000003	JMK133C	247KEB	1.16E+01	1.13E+01	1.12E+01	n/a	6.04E-05	94.8	3.70E-04

APPENDIX B

Chain of Custody Forms

K BASINS CHAIN OF CUSTODY

Chain of Custody No. 091495KE Date 09-14-95 Field Logbook No. N/A

OA&WH Contact M.A. GREEN Phone No. 373-1463 MSIN X3-67

Delivered to: ☐ OA&WH Engineer ☐ 183 KE Lab ☒ 222-S Lab ☐ Other

Sampled By SC OVERDAHL (Print) SC Overdahl (Sign)

See Sample Analysis Request for individual containers and analysis.

Sample Number	Location/Description	Sample Date	Sample Time	Matrix*	Comments
245 KEB	SA 213 / RW	9-14-95	1210	W	
246 KEB	SA 213 / RW	9-14-95	1210	W	
247 KEB	SA 213 / RW	9-14-95	1210	W	
248 KEB	SA 213 AFILTER / SND	9-14-95	1210	X	SAND

*Matrix

S	Soil
SE	Sediment
SO	Solid
SL	Sludge/Slurry
W	Water
O	Oil
A	Air
AF	Air Filter
DS	Drum Solids
DL	Drum Liquids
T	Tissue
WI	Wipe
L	Liquid
V	Vegetation
X	Other

Special Instructions:

RADIOACTIVE

Samples Transferred to new COC: ☒ No ☐ Yes, new COC No. _____

CHAIN OF POSSESSION

Relinquished By			Received By		
SC OVERDAHL	9/15/95		M.A. Green	9-15-95	
Print / Sign: SC Overdahl	Date	1250 Time	Print / Sign: M.A. Green	Date	1250 Time
M.A. Green	9-15-95		C Ed Byrd	9-15-95	
Print / Sign: M.A. Green	Date	1250 Time	Print / Sign: C Ed Byrd	Date	12:50 Time
C Ed Byrd	9-15-95		EE Dubos	9-15-95	
Print / Sign: C Ed Byrd	Date	13:35 Time	Print / Sign: EE Dubos	Date	1335 Time
Print / Sign	Date	Time	Print / Sign	Date	Time

FINAL SAMPLE DISPOSITION

Disposal Method _____

Disposed By: _____ Print / Sign / Date / Time

APPENDIX C

Letter of Instruction

From: Standards and Requirements 95-2A100.319
Phone: 373-9596
Date: August 17, 1995
Subject: LETTER OF INSTRUCTION FOR K BASINS SANDFILTER BACKWASH LINE SAMPLES, REVISION 1.

To: G. L. Miller T6-06

cc:

D. B. Bechtold	T6-09	M. A. Jensen	X3-79
C. L. Bennett	X3-79	C. D. Lucas	X3-67
S. P. Burke	X3-74	A. D. Rice	T6-06
B. S. Carlisle	X3-71	T. L. Welsh	T6-07
G. M. Davis	X3-80	SNF Project Files	R3-11
M. A. Green	X3-67	CDP File/LB	X3-79
R. A. Harris	L5-01		

Reference: Memo, C. DeFigh-Price to Distribution, "Letter of Instruction for KE Basins Sandfilter Backwash Line Samples," dated June 6, 1995.

This letter and attachment constitute a complete revision of the referenced Letter of Instruction.

Samples of the material flowing into each of the 105 K Basin sandfilter backwash pits (SFBWP) during a backwash will be sent to the 222-S Analytical Laboratory approximately twice per year. Each shipment will contain at least three but as many as five 250 mL sample bottles. This work is to be charged to TCPN L11AL/Work Order E26262. This Letter of Instruction will serve as an interim Sampling and Analysis Plan for all K Basin sandfilter backwash line samples.

C. DeFigh-Price, Manager
Standards and Requirements

CONCURRENCES:

G. L. Miller, Program Support
222-S Analytical Operations

8/18/95
G. M. Davis, QA
Spent Nuclear Fuel Project

jek

Attachment

INSTRUCTIONS FOR ANALYSIS OF SANDFILTER BACKWASH SAMPLES AT K-EAST FUEL STORAGE BASIN

Data Quality Objectives

The Data Quality Objectives (DQO) are described in WHC-SD-SNF-TA-007 (Harris, 1995). The primary objective of the analytical phase of the measurements is to determine the plutonium and uranium content of the samples to the requirements indicated in Table 1. The units of the Minimum Detection Level (MDL) and Practical Quantification Limit (PQL) refer to the samples as they are received from the field. The values of the MDL and PQL applicable to actual aliquots will be different because the analyte concentrations may be increased in the drying process and/or decreased during the acid digestion process (See Instructions below.) The extrapolation of these parameters is the responsibility of the 222-S laboratory. The definitions of the parameters in the table are given in Harris, 1995.

Table 1. Analytical Parameters

	$^{239/240}$Pu	Uranium
MDL	4.2 μCi/L of sample	23 mg/L of sample
PQL	43 μCi/L of sample	231 mg/L of sample
Precision	± 25% (2 RSD)	± 25% (2 RSD)
Accuracy	± 25%	± 25%

Instructions

The samples (250 mL bottles) will be received by the laboratory and prepared for laboratory analysis using the instructions that follow. These instructions are a modified version of preparation procedures (Bechtold 1993) that were developed to analyze samples (Warner 1994) expected to be very similar in content to the subject samples.

During sample preparation, excess water shall be removed from the samples for analysis, if specified on the chain of custody (COC) form accompanying the samples. If the COC makes no mention of this, then excess water is not to be collected or analyzed. The water samples will be analyzed for the alpha activity using the procedure shown in Table 2. It is expected that the plutonium content of the samples will be similar to that of the samples analyzed under Hunacek, 1994.

The primary analyses for the plutonium ($^{239/240}$Pu activity) and uranium content of the samples will utilize the procedures shown in Table 3. Alternate

procedures can be substituted for those shown in both Tables 2 and 3 if approved by signature of the manager, Standards & Requirements.

Table 2. Excess Water Procedure

Process	Constituent	Procedure ID
Internal Proportional Counter	Total α activity	LA-508-101

Table 3. Analytical Procedures and Process Requirements for 222-S Laboratory Primary Analyses

Process	Constituent	Procedure ID
Separation, AEA	^{238}Pu, $^{239/240}Pu$	LA-943-127
Laser Fluorimetry	U Total	LA-925-009

Analytical Quality Assurance Requirements

The Quality Assurance (QA) requirements for the final, primary analysis of the samples are given in Table 1. The QA requirements for the sample preparation phase are listed in the following sections.

There are no requirements on the analytical parameters (MDL, PQL, precision, accuracy) for special excess water analyses since the results will be used only for qualitative screening purposes.

Precision assessments using laboratory triplicates are required for *each sample*. The triplicates will be taken from acid digestates resulting from the performance of the following instructions. The assessment is made by computing the standard deviation of the three triplicate samples, dividing by the mean of those three samples, and then multiplying by 2 x 100. If the value obtained is greater than 25, the assessment must be rerun. If an acceptable value is not obtained after the rerun, contact Operations Analysis and Waste Handling (OA&WH) for further direction. Every attempt should be made to obtain valid results for each of the field samples. This is because the uncertainty used in the K Basins Process Standard C-303 verification analyses will be based solely on the three field samples.

Matrix spike samples for $^{239/240}Pu$ and uranium will be prepared and analyzed for each batch of samples processed. These samples require the addition of a known quantity of the analytes to the sample to measure analytical accuracy, and shall be created from the same digestate preparations used for triplicate analyses. If a spike recovery analysis differs from the expected value by more than the 25% limit, only a new spike sample will be created and analyzed

(not the entire batch). If the rerun does not produce an acceptable recovery, OA&WH will be notified. OA&WH will then specify the remedial action that will be taken by the laboratory.

Laboratory data will be maintained as NQA-1 or equivalent life-of-plant (K-Basins) QA records.

Reporting Requirements

The sample preparation results will be reported on a form similar to Table 4. They will be forwarded to OA&WH within four weeks of receipt of the samples. The values shall be reported in the units shown in Table 4.

The primary analytical results will be forwarded to OA&WH by electronic means within four weeks of receipt of the samples. The electronic file will be in the comma delimited format. Signed reports will be forwarded to OA&WH within six weeks after receipt of the samples. The units of the reported results should be per mL of the digestate produced by the following instructions. The results for each of the triplicate samples will be provided. Only descriptions of the matrix spike runs and the resulting accuracy comparisons are required.

General Preparation Instructions

The following instructions will be used to prepare samples obtained from the backwash line at both K East and K West Fuel Storage Basin for analysis at the 222-S laboratory. These instructions are based on the premise that the samples will closely resemble the samples collected in 1993 (Bechtold 1993.) That is, they will consist of heavy sand underlying flocculent sludge in a water medium. The water and heavy material are not expected to contain significant amounts of the materials of interest. Special measurements to confirm the content of the water will be included as an option until no longer required. The special measurement will be performed only if requested on the chain of custody (COC) form accompanying the samples.

In general, the sample preparation steps that evolved with the 1993 analyses will be followed (Bechtold 1993.) These steps are:

- remove the excess water over the settled sludge and dry it,
- acid digest the dried sludge, and
- sub-sample the digestate for the required component analyses.

It is very important to determine the weight and volumes of the materials and vessels both before and after each step in this preparation so that the final results can be related back to the original sample volume. Some exceptions to the 1993 test procedure were necessary. The major ones are listed below.

- No consolidation of a sample into a single container will be required because each will arrive at the laboratory in a single bottle.

- The volume of the original sample must be recorded because the laboratory results must be converted to mL of the original sample rather than to mL of sludge.

These exceptions require minor but numerous changes to the test procedure steps. Rather than refer to the 1993 test procedure and indicate each change, the steps appropriate for these analyses have been extracted. If there is any confusion about the meaning or context of these extracted steps (given below) the analyst should refer to the original (Bechtold 1993.)

The data that must be recorded during the preparation of the samples are summarized on Table 4. The data should be reported on this form or a similar one containing the information indicated.

Table 4. Sample Preparation Data

Step	Parameter Description		Sample Results			
1	Sample identity					
	amount of sludge	mL				
	contact dose rate	mR				
3	Gross weight of sample bottle	g				
9	Net weight of excess water (if requested)	g				
	Net weight of laboratory water sample	g				
	water sample number					
10	Gross wt. of sample bottle w/o excess water	g				
11	Tare weight of PTFE drying/digesting vessel	g				
15	Tare weight of sample bottle	g				
17	Weight of bottle filled to sludge mark	g				
19	Wt. of bottle filled to initial liquid level	g				
20	Gross wt. of drying vessel, first drying	g				
	second drying	g				
	third drying	g				
24	Net weight excess dry solids	g				
25	Gross weight solids to be digested	g				

Step	Parameter Description		Sample Results			
40	Volume of diluted digestate	mL				
44	Gross weight of dry digester plus residue	g				
	second digestion	g				
	third digestion	g				
46	Appearance of dried residue					

The precision and accuracy requirements for the data collected with these instructions (listed in Table 4) are given in Table 5.

Table 2. Data Precision and Accuracy Requirements

Datum	Precision	Accuracy
Sludge Observations	qualitative	qualitative
sludge height mark(s)	±10% of total height	±10% of total height
initial water level mark (original sample volume)	±3% of total height	±3% of total height
all weights (dry and liquid)	±0.1 g (scale accurate to 0.025 g)	5.0 g
digestate volume	volumetric glassware precision	volumetric glassware accuracy

PROCEDURE STEPS

"Drying to constant weight" in the following steps means attaining two successive weights whose difference is no more than 0.1% of the lowest calculated net weight of the two.

Hold points in the following steps are documented as released by a logged statement "Step __ released" followed by the signature of the responsible scientist.

Perform the following steps for each sample, recording all data and observations in a controlled laboratory notebook.

1. Examine all samples as-delivered by temporarily loading them one at a time into a radioactive service hood. Record each sample identity, approximate amounts of sludge, and contact dose rates in order to judge the need for hot cell facilities and to size the needed labware.

2. Load the sample into the hood or hot cell, whichever is appropriate, recording the sample identity.

3. Weigh and record the gross weight of the sample bottle.

4. Mark the sample bottle indelibly at the top of the liquid level. This is an important measurement that directly affects the ultimate use of the laboratory data. The bottle must be level and the mark must be made planar with bottom of meniscus. Note that the configuration of the bottle for this measurement must be as close as possible to that used in step 18.

5. Allow the sample bottle contents to settle in a level spot for a minimum of 24 hours and record the actual settling time.

6. Observe the sample bottle contents to verify the existence of a discernable boundary between the water and sludge.

NOTE: Step 7 is a HOLD POINT to await the determination of a water/sludge boundary.

7. Mark the sample bottle indelibly at the top of the sludge.

8. Vacuum suction off the excess water above the sludge in the sample bottle to a separate container without removing any sludge.

9. If specifically requested on the COC form, record the total weight of the excess water and create, weigh, and number a sample of the excess water. Perform a total alpha analysis on this sample. Retain the remaining excess water. Do not retain the excess water unless sampling was requested on the COC form.

10. Weigh and record the weight of the sample bottle without the excess water.

11. Weigh and record the tare weight of a suitably sized polytetrafluoroethylene (PTFE) drying/digesting vessel.

12. Quantitatively transfer the sample bottle's contents to the drying vessel, using a stirrer and rinsing with laboratory deionized water as necessary.

13. Place the drying vessel on a clean hot plate, along with a ventilating cover over the drying vessel to prevent dirt settling into it, and commence drying at a heat setting which produces approx. 120°C temperature at the contents when they are dry for at least 2 hours.

14. While drying the sludge solids, rinse clean and dry the empty sample bottle.

15. Weigh and record the tare weight of the empty sample bottle.

16. Add laboratory deionized water to the sludge height mark made in step 7.

17. Weigh and record the weight of the sample bottle plus laboratory deionized water.

18. Add laboratory deionized water to the initial liquid level mark made in Step 4. Be sure to have bottle level, and bring the bottom of the meniscus planar to the mark.

19. Weigh and record the weight of the sample bottle plus laboratory deionized water.

20. After drying the drying vessel and its contents for a suitable time interval (at least two hours), cool, reweigh and record the weight of the drying vessel and contents.

21. Thoroughly mix the drying vessel contents with the stirrer.

22. Repeat Steps 20. and 21. at least once, and as many times as necessary to come to a constant weight.

23. Obtain a suitable, labeled storage jar.

NOTE: Step 24 is a HOLD POINT to determine if there are sufficient dried solids present to divide into two portions.

24. Weigh out of the dryer/digester the excess dry solids over approximately 10 grams, if any, putting the excess into the storage jar, and leaving up to approximately 10 grams in the dryer/digester to be digested.

25. Record the new gross weight of the solids to be digested in the dryer/digester.

26. Obtain a suitable number of centrifuge cones with caps for use in clarifying digestates.

NOTE: Approximately 10M aqueous HCl may be conveniently prepared by carefully mixing concentrated hydrochloric acid reagent and laboratory deionized water at the rate of 1 mL acid per 0.245 mL water.

NOTE: Approximately 10M aqueous HNO_3 may be conveniently prepared by carefully mixing concentrated nitric acid reagent with laboratory deionized water at the rate of 1 mL acid per 0.6 mL water.

27. Prepare a digesting reagent consisting of (by volume) 1 part approximately 10M aqueous HCl and 1 part approximately 10M aqueous HNO_3.

28. Select a volumetric flask large enough to accommodate all clear digestate and subsequent rinsings of Steps 29. through 40., but no larger than 2 Liter capacity.

29. Add digesting reagent to the digesting vessel plus dry solids at the minimum rate of 2 volumes reagent per volume dry solids, but no more

than the digesting vessel can comfortably handle, and can be quantitatively transferred later.

30. Add concentrated HF to the digesting vessel at the rate of 1-2 drops concentrated HF per 100 mL of digesting reagent added.

31. Digest the solids by heating to near-boiling on the hot plate for four hours with a ventilating cover over the digester, adding digesting reagent as necessary to maintain volume.

32. Allow the digestate to cool and settle.

33. Decant the digester liquid, as much as will drain from the residues, into the cones.

34. Rinse the residues three times with acid, decanting the rinses into the cones each time.

35. Centrifuge the digestate/rinsates for 15 minutes.

36. Decant the clarified digestate into the volumetric flask, being careful to retain any residues in the cones.

37. Perform the following lettered sequence three times to rinse the cones:

    a. Rinse down the cones with acid.

    b. Centrifuge 15 minutes.

    c. Decant into the volumetric flask.

38. Mix the volumetric flask contents.

39. Rinse the residue from the cones back into the digester, using fresh digestion reagent as necessary, and Repeat Steps 29. through 38. to effect a second digest.

CAUTION: Addition of water to the digestate will generate large amounts of heat, enough to cause bumping in the volumetric flask if care is not taken. Add water slowly.

40. Slowly add laboratory deionized water to the volumetric flask while mixing by swirling, dilute to volume, thoroughly mix, and record the volume.

41. Rinse the residue from the cones back into the digester using laboratory deionized water as necessary.

42. Heat the digester with a ventilating lid on the hot plate to drive off the liquid and dry the residue at least two hours thereafter, using a setting which will produce approximately 120°C temperature in the dry residue.

43. Cool the digester.

44. Weigh and record the gross weight of the dry digester and residue.

45. Repeat Steps 42. through 44. at least once, and as many times as are required to come to a constant gross weight.

46. Observe and record the appearance of the dried residue. Dispose of the dried residue.

47. Withdraw a labeled, recorded aliquot of the volumetrically diluted digestate and submit to 222-S Analytical Operations for analyses.

48. Label and record the remainder of the volumetrically diluted digestate. This sample will be retained until released by K-Basin Operations.

49. Store all labeled portions of dry solids, digestate and dry residue.

50. Clean the work space, prepare reagents and equipment for the next sample.

REFERENCES

R. A. Harris, "Surveillance and Prediction Methods for the Plutonium Limit in the K East Fuel Storage Basin Sandfilter Backwash Pit," WHC-SD-SNF-TA-007, Revision 0, dated 1995.

G. S. Hunacek, Jr., "105 KE Fuel Storage Basin, Sampling and Analysis Plan," WHC-SD-NR-PLN-014, Revision 0, dated 1994.

R. D. Warner, "Safety Evaluation of the Plutonium and Uranium Content of the K East Basin Sandfilter Backwash Pit," WHC-SD-WM-TA-152, Revision 0, dated 1994.

D. B. Bechtold, "Laboratory Test Plan for Analysis of KE Basin Backwash Pit Samples," WHC-SD-NR-TP-023, Revision 1, dated 1993.

D. B. Bechtold, "Laboratory Test Plan for Analysis of KE Basin Backwash Pit Samples," WHC-SD-NR-TP-023, Revision 1, dated November 18, 1993.

DISTRIBUTION SHEET

To Distribution	From Production Planning and Control	Page 1 of 1 Date: 12/20/95
Project Title/Work Order WHC-SD-SNF-DP-006, Rev. 0, "105-K East Sandfilter Backwash Line Sample Analysis Report - Third Campaign"		EDT NO.: 614765 ECN NO.: N/A

Name	MSIN	Text With all Attach	EDT/ECN ONLY
Westinghouse Hanford Company			
R. B. Baker	L5-01	X	
C. L. Bennett	X3-79		X
S. T. Burke	X3-74	X	
C. Defigh-Price	X3-79	X	
J. L. Deichman	T6-03	X	
M. A. Green	X3-67		X
R. A. Harris	L5-01	X	
M. A. Jensen	X3-79	X	
C. D. Lucas	X3-67	X	
G. L. Miller	T6-06	X	
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