

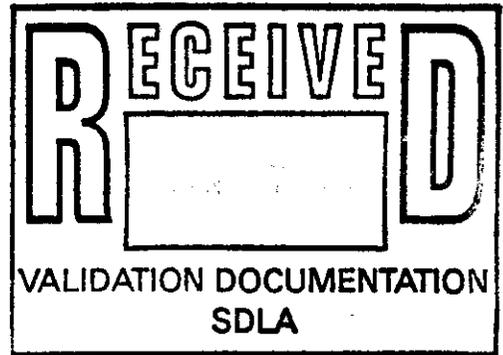
WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

VALIDATION SUMMARY

FOR

241-AP-107

WESTINGHOUSE 222-S LABORATORY



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This document has under gone two or three pagination processes. Two are from WHC, one is from PNL and one is from HASM.

The different formats are identified as follows:

- |    | Number Series  |
|----|--|
| 1) | WHC 1, 2, 3 etc  |
| 2) | WHC 1A-1, 1A-2, etc (for 222-S & PNL Addendums to original Document) |
| 3) | HASM 000006, 000007 etc  |
| 4) | PNL B01-001, B02-002 etc   |

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LABORATORY CASE NARRATIVE

WHC-SD-WM-DP-053  
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ADDENDUM 1A

List of Tables Within Tank 107-AP Case Narrative

- Table 1. Tank 107-AP Sample Sources and Identification
- Table 2. Laboratory Identification of Tank 107-AP Samples
- Table 3. Serial Number Extension Codes for Evaporator Samples
- Table 4. Maximum SW-846 Sample Holding Limits
- Table 5. Final Sample Volumes
- Table 6. TPP Cited Preparation Procedures vs. Actual Procedure Used
- Table 7. 107-AP Procedure Listing
- Table 8. Relevant Boundary Conditions for Tank 107-AP
- Table 9. Appearance/Homogeneity Analysis Summary
- Table 10. Summary of DSC Results
- Table 11. New ICP LMCS Data
- Table 12. Homogeneity Analysis, a Comparison of RPD Data
- Table 13. Summary of Analytical Data for Tank 107-AP

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ANALYSIS AND CHARACTERIZATION OF DOUBLE  
SHELL TANK 241-AP-107

CASE NARRATIVE

INTRODUCTION

SOURCE DOCUMENTATION

On August 1, 1993, evaporator feed tank 241-AP-107 (hereafter referred to as 107-AP) was sampled under the protocol described in 242-A Evaporator Waste Analysis Plan, WHC-SD-WM-EV-060, Rev. 2 and in 242-A Evaporator Quality Assurance Project Plan, WHC-SD-WM-QAPP-009, Rev. 0. Waste contained in tank 107-AP was characterized chemically by the Westinghouse Hanford Company 222-S Laboratory as directed by four documents:

1. 242-A Evaporator Waste Analysis Plan (WAP), WHC-SD-WM-EV-060, Rev. 2
2. 242-A Evaporator Quality Assurance Project Plan (QAPP), WHC-SD-M-QAPP-009, Rev. 0,
3. 242-A Evaporator Project Analytical Services, Statement of Work (SOW), (WHC-SOW-93-0006, Rev. 1, and
4. Technical Project Plan for the 222-S Laboratory in Support of the 242-A Evaporator Waste Analysis Plan, WHC-SD-WM-EV-060, Revision 2, WHC-SD-WM-TPP-048, Rev. 0, Technical Project Plan (TPP).

Laboratory operations at the 222-S Laboratory are performed according to the Quality Assurance Project Plan for the Analysis of Highly Radioactive Samples in Support of Environmental Activities on the Hanford Site, WHC-SD-CP-QAPP-002, unless superseded by the WAP, QAPP, SOW or TPP. Deviations from these documented instructions are discussed in this narrative and are generally supported with additional documentation.

Physical, inorganic, and radiochemical analyses were performed on tank 107-AP by the 222-S Laboratory. Organic analyses on this tank were performed by Battelle Pacific Northwest Laboratory (PNL) and are not discussed.

Characterization of evaporator feed tank waste is needed primarily for an evaluation of its suitability to be safely processed through the evaporator. Such analyses should provide sufficient information regarding the waste composition to confidently determine whether constituent concentrations are within not only safe operating limits (e.g., nonflammable, nonexplosive), but should also be relevant to functional limits (e.g., solids formation) for operation of the evaporator. Characterization of tank constituent concentrations should provide data which enable a prediction of where the types and amounts of environmentally hazardous waste are likely to occur in the evaporator product streams.

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## RELEVANCE TO SW-846 PROCEDURES

Although SW-846 methods are mentioned in the source documents as examples of the desired analytical technology to use in this characterization effort, it is clear that the evaporator program anticipated the use of in-house controlled procedures within the 222-S Laboratory. The WAP, section 5.1.2 states, "Laboratory operations and test procedures that are performed in carrying out the requested characterization activities will be determined by laboratory personnel and will be defined in the TPP".

Each inorganic chemical analyses performed by the 222-S Laboratory was conducted using a controlled procedure. Some procedures were consistent with SW-846 guidance and others are patterned after U.S. Environmental Protection Agency, Contract Laboratory Program (CLP) methods. All procedures, however, use SW-846 analytical technology. Deviations from exact SW-846 procedures (such as sampling, aliquot size, sample holding time and sample preservation) are essential when handling samples containing dangerous levels of radioactive material, and are consistent with the final rule regarding "Hazardous Waste Management System; Testing and Monitoring Activities, 40 CFR Parts 260, 261, 264, 265, 268, and 270 as stated in the Federal Register, volume 58, number 167, Tuesday, August 31, 1993. Generally these deviations are practiced to limit exposure of personnel to ionizing radiation to an amount that is "as low as reasonably achievable" (ALARA).

## SAMPLING

Tank 107-AP samples were collected on August 1, 1993 between 1005 hours and 1220 hours from three vertical risers within the tank. Duplicate samples were collected at each of the five sampling locations. Two sample locations were selected at random depths within the tank from risers 1(NW) and 1(E). The fifth sample location was within the remaining riser 1(SW).

The "bottle-on-a-string" method was used to collect the liquid samples from the tank. Each glass sample bottle contained approximately 100 milliliters and was closed with a teflon seal cap.

Duplicate field blanks were generated by placing deionized water into sampling bottles and closed with the same caps used for the tank samples.

Each sampling location and field blank was sampled in duplicate, therefore, one complete set of samples was available to both the 222-S Laboratory and to the PNL Laboratory. One additional sample was collected from the surface of the tank waste and provided to PNL for a Total Organic Carbon analysis.

Samples were not preserved (neither acidified nor refrigerated) nor was there an attempt to assure the complete filling of the bottles so as to exclude all headspace. These actions were consistent with safety procedures, which attempt to limit personnel exposure to hazardous ionizing radiation.

Chain-of-Custody forms were generated by the sample collector. Samples were transported to the laboratory receiving door in pigs by the B-Plant sample truck. All samples were received into the laboratory on August 2, 1993 at 1730 hours.

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**LABORATORY OPERATIONS****SAMPLE TRACKING****RECEIVING PROCEDURES/CHAIN-OF-CUSTODY**

Tank 107-AP samples were received into the laboratory at door 13, where the laboratory sample custodian signed the Chain-of-Custody form as the new sample custodian. The Chain-of-Custody form is a legal document, which tracks the transfer of samples between individuals or organizations to establish sample ownership.

The pigs containing the samples were transported to a hood. The radioactive dose rate "over the top" of each pig's opening was measured by a Health Physics Technician (HPT). It was determined that the dose rate for all samples was less than 2 mrem per hour. Samples were removed from the pigs, labeled with the laboratory identification number, and transferred to the metal storage cabinets in a secured area.

Subsequently, a determination was made to not process the samples through the hot cell, thus expediting the analyses. Hot cell processing of samples is required when the "over-the-top" dose rate exceeds 2 rem per hour or 25 rad per hour.

**REQUEST FOR SPECIAL ANALYSIS**

Request for Special Analysis (RSA) forms were generated for each sample, which provided a laboratory identification number, stated the preparative and analytical procedures, set groups of samples into analytical batches, and stated the degree of quality control. RSAs served as the controlling documents for analytical operations. Sample preparation instructions on the RSAs included such things as dilutions, acid digestions, and compositing. Hydroxide, arsenic, and tritium are examples of analytical procedures shown on the RSA sheet. Generally a maximum of three samples were included in a sample batch so that quality control determinations (such as a blank, standard(s), duplicate sample, spiked sample, and duplicate spiked sample) may be included. From the instructions on the RSA, traveler cards were generated.

**SAMPLE IDENTIFICATION**

The 107-AP samples were identified by the sample collector using a shipping number which began with "R". Table 1 was taken from a Process Memo on "Bottle on a String Sample of Tank 107-AP", dated July 21, 1993, which was generated by Double Shell Tanks. It references the shipping number to the sampling location within tank 107-AP. As can be seen in Table 1, duplicate samples from each location were given unique shipping numbers and were sent to either the Westinghouse Hanford Company's (WHC) 222-S Laboratory or to the Battelle Pacific Northwest Laboratory (PNL), also known as the 325 Laboratory.

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Table 1. Tank 107-AP Sample Sources and Identification				
Shipping Number	Riser	Sample Elevation (inches)	Sample Type	String Length (inches)
R3619	1(NW)	60	inorg/rad (WHC)	589
R3620		60	organic (PNL)	589
R3621		144	inorg/rad (WHC)	505
R3622		144	organic (PNL)	505
R3623	1(E)	204	inorg/rad (WHC)	445
R3624		204	organic (PNL)	445
R3625		24	inorg/rad (WHC)	625
R3626		24	organic (PNL)	625
R3627	1(SW)	228	inorg/rad (WHC)	421
R3628		228	organic (PNL)	421
R3910		228	organic (WHC)	421
R3629		402	surface sample toc/appr (PNL)	247
R3630	---	---	field blank inorg/rad (WHC)	---
R3631	---	---	field blank organic (PNL)	---

Upon arrival at the 222-S Laboratory, each sample was given a laboratory identification number, which began with "V", and a description.

Each of the five samples were analyzed for physical parameters and for inorganic constituents. A composite sample was generated from each of the five samples in equal proportions for radiochemical analyses.

Table 2 shows the relationship between the original sample number, the laboratory sample identification number, and the laboratory sample description.

The sample description was a letter designation, A through E, added as a suffix to the tank number. For example, "107-AP-A" served as an easy way to remember the sample identification for R3619 from riser 1(NW) 60 inches. It was shown on the RSA in the "Customer ID" column.

Shipping sample R3910 was collected and delivered to the 222-S laboratory for internal, non-characterization purposes, consequently data for this sample are not included in this data package.

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Table 1. Tank 107-AP Sample Sources and Identification				
Shipping Number	Riser	Sample Elevation (inches)	Sample Type	String Length (inches)
R3619	1(NW)	60	inorg/rad (WHC)	589
R3620		60	organic (PNL)	589
R3621		144	inorg/rad (WHC)	505
R3622		144	organic (PNL)	505
R3623	1(E)	204	inorg/rad (WHC)	445
R3624		204	organic (PNL)	445
R3625		24	inorg/rad (WHC)	625
R3626		24	organic (PNL)	625
R3627	1(SW)	228	inorg/rad (WHC)	421
R3628		228	organic (PNL)	421
R3910		228	organic (WHC)	421
R3629		402	surface sample toc/appr (PNL)	247
R3630	---	---	field blank inorg/rad (WHC)	---
R3631	---	---	field blank organic (PNL)	---

Upon arrival at the 222-S Laboratory, each sample was given a laboratory identification number, which began with "V", and a description.

Each of the five samples were analyzed for physical parameters and for inorganic constituents. A composite sample was generated from each of the five samples in equal proportions for radiochemical analyses.

Table 2 shows the relationship between the original sample number, the laboratory sample identification number, and the laboratory sample description.

The sample description was a letter designation, A through E, added as a suffix to the tank number. For example, "107-AP-A" served as an easy way to remember the sample identification for R3619 from riser 1(NW) 60 inches. It was shown on the RSA in the "Customer ID" column.

Shipping sample R3910 was collected and delivered to the 222-S laboratory for internal, non-characterization purposes, consequently data for this sample are not included in this data package.

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Table 2. Laboratory Identification of Tank 107-AP Samples

Original Sample Number	Lab Sample Number	Lab Sample Description
- - -	V19	LMCS Standard for V21, V23
- - -	V20	Reagent Blank for V21, V23
R3619	V21	107-AP-A
R3621	V23	107-AP-B
- - -	V24	LMCS Standard for V26 - V28
- - -	V25	Reagent Blank for V26 - V28
R3623	V26	107-AP-C
R3625	V27	107-AP-D
R3627	V28	107-AP-E
- - -	V29	LMCS Standard for V31
- - -	V30	Reagent Blank for V31
R3630	V31	Field Blank, 107-AP-FB
- - -	V32	LMCS Standard for V34
- - -	V33	Reagent Blank for V34
Composite of R3619, R3621, R3623, R3625 and R3627	V34	107-AP-COM
- - -	V45	LMCS Standard for V24 - V28
- - -	V46	LMCS Standard for V24 - V28
- - -	V47	LMCS Standard for V29 - V31
- - -	V48	LMCS Standard for V29 - V31
- - -	V49	LMCS Standard for V19 - V23
- - -	V50	LMCS Standard for V19 - V23
- - -	V51	LMCS Standard for V24 - V28
- - -	V52	LMCS Standard for V24 - V28
- - -	V53	LMCS Standard for V29 - V31
- - -	V54	LMCS Standard for V29 - V31
- - -	V57	LMCS Standard for V19 - V23
- - -	V58	LMCS Standard for V19 - V23
- - -	V59	LMCS Standard for V19 - V23

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Table 3. Serial Number Extension Codes for Evaporator Samples

Sample Preparation	Direct	Vxx.5xxx
	Water Leach (water dil'n)	Vxx.7xxx
	Acid Digest	Vxx.8xxx
Quality Control Function	LMCS Standard	Vxx.x5xx
	Reagent Blank	Vxx.x6xx
	Sample	Vxx.x7xx
	Sample Duplicate	Vxx.x8xx
	Spike	Vxx.x9xx
	Spike Duplicate	Vxx.x0xx
Analysis Type	Appearance	Vxx.xx01
	Specific Gravity	Vxx.xx06
	Differ. Scanning Calorimetry	Vxx.xx11
	Total Beta	Vxx.xx20
	Total Alpha	Vxx.xx25
	Total Inorganic Carbon	Vxx.xx27
	Ammonia	Vxx.xx28
	Hydroxide	Vxx.xx29
	Gamma Energy Analysis	Vxx.xx30
	Uranium, total	Vxx.xx40
	Inductively Coupled Plasma	Vxx.xx50
	Ion Chromatography	Vxx.xx71
	Cyanide	Vxx.xx78
	Pu <sup>239/240</sup>	Vxx.xx81
	Am <sup>241</sup>	Vxx.xx82
	Np <sup>237</sup>	Vxx.xx83
	Tc <sup>99</sup>	Vxx.xx84
	I <sup>129</sup>	Vxx.xx85
	Sr <sup>90</sup>	Vxx.xx86
	H <sup>3</sup>	Vxx.xx87
	C <sup>14</sup>	Vxx.xx88
	Se <sup>79</sup>	Vxx.xx89
	Arsenic	Vxx.xx95
	Selenium	Vxx.xx96
Mercury	Vxx.xx97	

No extension code number was generated for curium-244, because it does not have a specific procedure number. It is determined, however, when americium-241 is analyzed.

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When the codes for the evaporator project were generated on the computer, a code for a simple sample dilution was omitted because no controlled procedure for sample dilution was available. It was necessary to track all sample handling within the laboratory, including dilution factors, consequently the code for a water digestion (actually a water leach procedure for solid samples, LA-504-101) was used. It should be clearly understood that the tank samples were *not* subjected to a leaching procedure, but the traveler cards which were generated with the water digestion code were used simply to document the dilution factors acquired during the sample preparation phase.

### ANALYTICAL BATCH SHEET

An Analytical Batch sheet was prepared for each batch of traveler cards by a Lab Leader. Data were added to this sheet by the technician, who performed the analysis, which describe analytical conditions (e.g., analyst, instrument, date, time, temperature, etc.) and procedures used (e.g., type of sample preparation, analytical procedure, and revision numbers).

### ANALYTICAL BATCH SUMMARY SHEETS

Upon completion of each analytical batch, several data calculation or evaluation steps were performed. Raw data were converted to final values by technicians. Chemists reviewed the calculations for approval and then generated an Analytical Batch Summary Sheet for all analyses (except for inductively coupled plasma/emission spectrometry) (ICP). This sheet brought together the final data from each of the traveler cards (representing each determination). Additional information from calculations of quality assurance parameters, such as percent recovery, relative percent difference (RPD), etc., was added to the summary sheet by the chemists. Occasionally, chemists include descriptive information regarding the batch on these sheets. This information is included in this case narrative.

All data were reviewed by the chemists and the project coordinator to determine that correct values were transcribed from the traveler cards to the analytical batch summary sheets.

Instead of generating an Analytical Batch Summary Sheet for ICP, raw analytical data as well as analytical quality control parameters were summarized into an independent report known as the "Ward's<sup>1</sup> package". It was necessary to use this Ward's package in place of the batch summary sheet because of the significantly greater quantity of data generated by ICP analyses.

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<sup>1</sup> Ward's is a trademark of Ward's Scientific, LTD.

**ANALYTICAL AND REPORT SCHEDULING**

A computerized logic schedule was generated to plan each major event necessary to produce the final data package. Assignees were assigned to each event. During the weekly scheduling meeting, each assignee was responsible for reporting progress on assigned actions. Milestones were closely monitored to assure that targeted schedule dates were being met. Reports, which tracked the scheduling status, were published and distributed weekly throughout the WHC Processing and Analytical Laboratories division.

A preliminary report of data through the use of the LCCS was scheduled by the Statement of Work (SOW) to be due to the customer (the evaporator program) 85 days after collection of the last sample from tank 107-AP. That date was October 25, 1993. In subsequent discussions with the customer, it was mutually agreed that an LCCS report would not be required, if instead the data package (as requested in the SOW) could be provided to the customer (and to Hanford Analytical Services Management [HASM]) on or about the same date that the LCCS report would have been due. It was also understood that the package delivered to the customer at that time would not be a validated package, because validation would be performed by HASM.

An electronic mail message sent to the 222-S project coordinator from the customer (John F. O'Rourke, memo dated on November 19, 1993) is evidence of this agreement. This document is shown in the Communication Documents section.

**QUALITY ASSURANCE OF THE ANALYTICAL SYSTEM AND DATA****CONTROLLED PROCEDURES**

Every analytical procedure used to develop data for the evaporator project was performed under the direction of written procedures (except for the dilution procedure) which are reviewed and approved at least every two years. Revisions to existing procedures were and are generated for corrections or updating as needed, and are distributed promptly to replace obsolete versions.

**STATISTICAL EVALUATION**

Performance data have been gathered historically on each analytical procedure for which there is a Laboratory Measurement Control System (LMCS) standard available. The percent recovery (an assessment of procedure accuracy) has been determined and stored in the LMCS computer data base for each LMCS standard. On a yearly basis, a statistical evaluation of these data has been made to generate new acceptance control limits for future analyses of LMCS standards.

An evaluation of the mean and standard deviation values for each of the procedures has also been used to signal needed improvements in these procedures, when means deviate significantly from 100 percent or when standard deviations are relatively large.

**REVIEW OF DATA**

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Each batch has been reviewed for accuracy at several levels. Chemists reviewed not only analytical calculations, but checked to determine that the

analytical system was performing appropriately and that the laboratory technicians were following written procedures.

A team of technicians recalculated values on the traveler cards, and checked the information reported by the chemists for completeness.

The Quality and Technology Services department of PAL performed the second general review of analytical data. They recalculated final values from the raw data as well as checked the information reported by the chemists and technicians for completeness.

A third general review was performed by the project coordinator, which included those items checked previously by others, but also included spreadsheet summaries of data. A peer review of this narrative was performed by other project coordinators in the PAL, Analytical Operations, Program Support Group.

The Quality and Technology Services group within PAL has performed a topical review of the data package.

## **ANALYTICAL DATA REPORTING**

### **LABORATORY CUSTOMER COMPUTER SYSTEM**

Following the completion of Analytical Batch Summary Sheets, all data per batch are taken to a Lab Leader for keyboard entry of final values into the Laboratory Customer Computer System (LCCS).

### **ANALYTICAL DATA SPREADSHEETS**

Spreadsheets were generated in a tabular summary format to facilitate evaluation of both final and quality control data. All data were reviewed by the project coordinator to determine that these data were transcribed correctly from the batch summary sheets and Ward's™ package to the spreadsheets. These spreadsheets are incorporated into the Results of Analyses section of this narrative.

## **CASE NARRATIVE**

This case narrative was prepared in accordance with the following source documents:

- WAP, section 5.1.7,
- QAPJP, sections 3.1, 3.2, 3.4, 3.6, 7.1, 7.3, and 8.1 (with the exception that the "QA Report" that was generated was not produced by a QA Officer),
- SOW, sections 4.0 through 4.3, and
- TPP, sections 2.2.4, 5.1 and 5.3.

The intent of this narrative is to:

- Present required analytical data
- Evaluate the quality of these data
- Document problems with procedures or the data generated from these procedures (including quality control data)
- Characterize the nature of the constituents within tank 107-AP, and
- Interpret, whenever possible, the relevance or impact of these findings on the evaporator program.

**SAMPLE PRESERVATION**

No preservation of samples (acidification or refrigeration) was performed at the time of sampling as discussed in the WAP, section 5.1.5.1.

No attempt was made to preserve the samples while they were held in the laboratory because of the small initial sample volume (100 ml). The project coordinator was concerned that if part of the sample was split for acid preservation, that likely there would be an insufficient sample volume remaining to perform all of the requested analyses. See the discussion below on Analytical Aliquots for further details.

**SAMPLE HOLDING TIME**

The QAPJP, section 3.6 states the following:

"Holding times for samples that need not require processing in the hot cell shall be equivalent to SW-846 (EPA 1986) defined times. If no SW-846 holding time exists for a specific constituent, the holding time will be three months."

SW-846 maximum sample holding time limits, as stated in volume I, section A, part 2.6 (revision 1, July 1992), for analytes specific to this project are shown in Table 4.

Table 4. Maximum SW-846 Sample Holding Limits		
Parameter	Maximum Holding Time	Preservation
Chloride	28 days	none
Cyanide	14 days	4°C
Nitrate	48 hours	4°C
Sulfate	28 days	4°C
Mercury	38 days (in glass)	HNO <sub>3</sub> to pH<2
Metals *	6 months	HNO <sub>3</sub> to pH<2
Total Alpha	6 months	HNO <sub>3</sub> to pH<2
Total Beta	6 months	HNO <sub>3</sub> to pH<2
Radium **	6 months	HNO <sub>3</sub> to pH<2

\* Other than Hg & Cr<sup>+6</sup>

\*\* Radiological

Agreement within the scientific community is divided with regard to reasonable sample holding times. SW-846 holding times are based on worst-case scenarios and in many cases are excessively short. Note that SW-846 protocol expects samples are preserved at the time of collection for SW-846 holding times to be valid. However, Tank 107-AP samples were intentionally not preserved to limit the exposure dosage of ionizing radiation to personnel.

Sample degradation can occur due to many factors. One of these factors, biological degradation, is typically controlled by the addition of a strong acid, creating a hostile biological environment due to extreme pH. For biological degradation of tank 107-AP samples, it can be argued that sample



Table 6 shows a comparison of the preparative procedures stated in the TPP with preparative procedures actually performed:

Table 6. TPP Cited Preparation Procedures vs. Actual Procedure Used		
Analytical Procedure	Procedure Stated in TPP	Actual
Appearance	direct	direct
Diff. Scanning Calorimetry	direct	direct
Specific Gravity	direct	direct
Induct Coupled Plasma	acid digestion	acid digestion
Arsenic	acid digestion	acid digestion
Selenium	acid digestion	acid digestion
Mercury	direct	direct
Uranium	direct	direct
Ion Chromatography	direct or H <sub>2</sub> O dilution	H <sub>2</sub> O dilution
Ammonia	direct or acid dilution	direct
Cyanide	direct or H <sub>2</sub> O dilution	H <sub>2</sub> O dilution
CO <sub>3</sub> (TIC)	direct or H <sub>2</sub> O dilution	direct
Hydroxide	direct or H <sub>2</sub> O dilution	direct
Gamma Energy Analysis	acid dig, direct, or acid dil	acid digest
Pu <sup>239/240</sup>	acid dig, direct, or acid dil	acid digest
Am <sup>241</sup>	acid dig, direct, or acid dil	acid digest
Cm <sup>244</sup>	acid dig, direct, or acid dil	acid digest
Np <sup>237</sup>	acid dig, direct, or acid dil	direct
Sr <sup>90</sup> *	direct or acid dilution	acid digest
Tc <sup>99</sup> *	direct or acid dilution	acid digest
Se <sup>79</sup> *	direct or acid dilution	acid digest
C <sup>14</sup>	direct or H <sub>2</sub> O dilution	direct
H <sup>3</sup>	direct or H <sub>2</sub> O dilution	direct
I <sup>129</sup>	direct or H <sub>2</sub> O dilution	direct
Total Alpha *	direct or acid dilution	acid digest
Total Beta	acid dig, direct, or acid dil	acid digest

\* Deviations from stated procedure in TPP.

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# ANALYTICAL PROCEDURES

APPENDIX 1A

Each procedure used for this project was a controlled procedure. All procedures were evaluated and approved for a maximum period of two years. Procedures may be modified, or deleted as appropriate. Each time a procedure was modified, however, a new revision number was added to the procedure number. Upon review (at the end of the two year period), a procedure approval may be extended for another two year period without a change in revision number. During this project the following procedures were reviewed, approved, and extended without a revision number change.

Strontium-90	LA-220-101/D-0	Reviewed 6/22/93
Specific gravity	LA-510-112/C-2	Reviewed 6/26/92
Diff. scan. calorim.	LA-514-113/A-0	Reviewed 7/8/93
Gamma energy analysis	LA-548-121/D-0	Reviewed 6/9/93

Analytical procedures used for the evaporator project were listed initially in the TPP and are shown in Table 7. Several of those listed in the TPP were for process control sample analyses (nonprotocol) and were not used for the evaporator feed tank samples.

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Table 7. 107-AP Procedure Listing

TPP Cited Procedure		Actual Procedure Used				
Procedure #	Procedure Title	Procedure #	Rev #	Procedure Title	Issue Date	Analysis Dates
LA-505-151	ICP Emission Spectrometer Method for Trace Element Analysis	LA-505-151	D-0	ICP Emission Spectrometer Method for Trace Element Analysis	02-17-93	
LA-325-104	Mercury Analysis by Atomic Absorption (Manual Cold Vapor Technique)	LA-325-104	A-1	Mercury Analysis by Atomic Absorption (Manual Cold Vapor Technique)	03-12-93	09-05-93 09-06-93
LA-695-102	Microdistillation and Spectrophotometric Determination of CN	LA-695-102	B-0	Microdistillation and Spectrophotometric Determination of CN	10-03-91	08-30-93 08-31-93
LA-365-132	Determination of Se-79	LA-365-132	B-0	Determination of Se-79	09-25-91	09-02-93
LA-510-112	Specific Gravity of High Beta Gamma Caustic Samples	LA-510-112	C-2	Specific Gravity of High Beta Gamma Caustic Samples	Reviewed 06-26-92	08-22-93
LA-519-151	Visual Check and Over-The-Top Reading	LA-519-151	E-1	Visual Check and Over-The-Top Reading	02-22-93	08-10-93
LA-533-101	Anion Analysis on Dionex Model 10	Not Used		See LA-533-105		
LA-355-131	Arsenic Analysis by Hydride Generation Atomic Absorption	LA-355-131	B-1	Arsenic Analysis by Hydride Generation Atomic Absorption	12-09-92	09-01-93 09-02-93 11-04-93 11-09-93
LA-365-131	Selenium Analysis by Hydride Generation Atomic Absorption	LA-365-131	B-2	Selenium Analysis by Hydride Generation Atomic Absorption	12-09-92	08-24-93 09-07-93 09-08-93
LA-514-113	Differential Thermal Analysis of Caustic Samples	LA-514-113	A-0	Differential Thermal Analysis of Caustic Samples	Reviewed 07-08-93	08-31-93 09-01-93 09-09-93
	Not Cited	LA-505-158	A-2	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA and ICP Spectroscopy	07-31-93	08-11-93 08-16-93
	Not Cited	LA-505-158	A-3	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA and ICP Spectroscopy	09-14-93	10-11-93 11-02-93
	Not Cited	LA-504-101	C-0	Water Leach of Solids with Residual Solids Collection.	06-23-93	08-10-93
LA-548-121	Preparation of Sample Mounts for GE(LI) GEA - Low Level	LA-548-121	D-0	Preparation of Sample Mounts for GE(LI) GEA - Low Level (preparation for LA-508-052)	07-10-91	
	Not Cited	LA-508-114	A-1	Operation of Gamma Products Alpha Beta Counting Systems Using PC Control (Subsequent to LA-508-101)	Reviewed 05-15-92	

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Table 7. 107-AP Procedure Listing

TPP Cited Procedure		Actual Procedure Used				
Procedure #	Procedure Title	Procedure #	Rev #	Procedure Title	Issue Date	Analysis Dates
LA-508-101	Low Level Alpha and Beta in Water Samples (prep)	LA-508-101	D-0	Low Level Alpha and Beta in Water Samples (preparation for LA-508-114)	08-19-92	08-24-93 09-01-93 09-08-93 10-04-93 10-25-93
LA-503-156	Determination of Pu and Ion Exchange Solvent Extraction	LA-503-156	D-0	Determination of Pu and Ion Exchange Solvent Extraction (also Am-241 and Cm-244)	12-03-91	09-01-93 09-02-93 09-08-93
LA-933-141	Determination of Np-237 by ToIOA/TTA Extraction and Alpha Counting	LA-933-141	H-0	Determination of Np-237 by ToIOA/TTA Extraction and Alpha Counting	09-09-91	09-02-93
LA-220-101	High Level Strontium-89, 90 in Aqueous Samples	LA-220-101	D-0	High Level Strontium-89, 90 in Aqueous Samples	Reviewed 06-22-93	09-20-93
LA-438-101	Determination of Tc-99 by Solvent Extraction and Liq. Scint Counting	LA-438-101	D-1	Determination of Tc-99 by Solvent Extraction and Liq. Scint Counting	09-04-91	09-01-93
LA-378-103	Determination of Iodine-129 in Waste Tank Samples	LA-378-103	B-2	Determination of Iodine-129 in Waste Tank Samples	06-28-93	08-11-93
LA-348-104	C-14 in Small Volume Samples by Persulfate Oxidation and Liq. Scint.	LA-348-104	B-0	C-14 in Small Volume Samples by Persulfate Oxidation and Liq. Scint.	07-31-91	10-14-93
LA-218-114	Tritium by Lachat Micro-Dist. and Liquid Scintillation Counting (LS)	LA-218-114	A-2	Tritium by Lachat Micro-Dist. and Liquid Scintillation Counting (LS)	01-14-93	08-17-93 10-15-93
LA-508-121	Operation of the Beckman Liquid Scintillation Counter	LA-508-121	B-0	Operation of the Beckman Liquid Scintillation Counter (subsequent to LA-218-114 & LA-348-104)	11-25-91	
LA-533-105	Anion Analysis of Dionex Model 4000i	LA-533-105	C-0	Anion Analysis of Dionex Model 4000i	02-12-93	08-11-93 08-12-93 08-16-93
LA-533-105	Anion Analysis of Dionex Model 4000i	LA-533-105	C-1	Anion Analysis of Dionex Model 4000i	Reviewed 08-24-93	08-30-93 09-07-93
LA-925-106	Determination of Uranium by Laser Fluorimetry	LA-925-106	B-0	Determination of Uranium by Laser Fluorimetry	02-18-92	09-09-93 09-13-93 09-22-93 10-27-93 11-01-93
LA-645-001	Spectrophotometric Determination of Nitrite	Not Used		See LA-533-105		
LA-212-101	Set Up and Standardization of pH Meter and Glass Electrode	Not Used		See LA-211-102		
LA-212-102	Determination of pH Direct Measurement	Not Used		See LA-211-102		

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Table 7. 107-AP Procedure Listing

TPP Cited Procedure		Actual Procedure Used				
Procedure #	Procedure Title	Procedure #	Rev #	Procedure Title	Issue Date	Analysis Dates
LA-661-102 or LA-661-103	Determination of Hydroxide Ion in solutions containing Hydrolyzable Anions (Auto) Determination of OH Ion in Solutions by Potentiometric Titration Manual	LA-211-102	B-0	Determination of Acid/Base/pH Using Metrohm 682 Titroprocessor (Hydroxide)	04-22-93	08-11-93
LA-634-102	Ammonia by Kjeldahl	LA-634-102	D-1	Ammonia by Kjeldahl	06-02-93	08-20-93 08-30-93 08-31-93
LA-622-102	Determination of Carbonate in Solutions by Coulometry	LA-622-102	B-2	Determination of Carbonate in Solutions by Coulometry	04-14-92	08-10-93 08-11-93 08-14-93 08-20-93
LA-508-052	Gamma Energy Analysis on the Canberra Jupiter System	LA-508-052	B-4	Gamma Energy Analysis on the Canberra Jupiter System (subsequent to LA-548-121)	Reviewed 06-09-93	

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## DETECTION LIMITS

Detection limits were defined for each procedure without reference to a uniform laboratory protocol to determine such limits. ICP detection limits were derived from CLP protocol. Some of the procedures used the reagent blank value as the detection limit. Some procedures used the concentration of the lowest standard in the calibration curve as the detection limit. In no case was the matrix considered in generating the detection limit and therefore would not qualify as a "method detection limit". Wherever possible, and unless stated differently in the discussion of each analyte, the detection limit was modified by the typical dilution factor of the samples to provide a more representative value relative to the samples. All of those practices described above are allowed as estimated quantitation limits by SW-846 protocol.

## SIGNIFICANT FIGURES

The QAPjP, page 6, states that the report must not contain a greater number of significant figures than is defined in the procedure. A review was made of each controlled procedure to insure compliance with any stated significant figure requirements. The only procedure which specified significant figures was specific gravity. For this parameter, the reported value must have three digits to the right of the decimal in standard numerical notation (not scientific notation) format. Specific gravity was reported with the specified number of significant figures.

## CALIBRATION DATA

The QAPjP states that for manually generated calibration curves, sufficient data will be placed into the data package that will allow the recalculation of the slope, Y-intercept, and correlation coefficient ( $r^2$ ); and that the  $r^2$  is within specifications given in the procedure.

Raw calibration data were available on the analytical traveler cards or summary sheets and are not given in this narrative. These cards are incorporated into the data package as an appendix for reference. For those parameters that a standard curve was manually generated, the slope, Y-intercept, and correlation coefficient ( $r^2$ ) are provided in the Results of Analyses section of the narrative.

Only two of the 222-S analytical procedures stated a required correlation coefficient ( $r^2$ ). The required correlation coefficient for mercury is  $r^2 > 0.995$ . Cyanide has a required  $r^2$  of  $> 0.999$ . During analyses, both cyanide and mercury had an  $r^2$  value greater than the required value for all batches.

## EVAPORATOR BOUNDARY CONDITION LIMITS

To evaluate compliance with required boundary conditions, final values for Differential Scanning Calorimeter, nitrate, nitrite and hydroxide concentrations have been used. These are shown and evaluated in the Results of Analyses section and in the Summary Tables.

Those boundary conditions given in section 4 of the Waste Analysis Plan which were relevant to the analyses performed by the 222-S laboratory are shown in Table 8.

Table 8. Relevant Boundary Conditions for Tank 107-AP	
Parameter	Boundary Condition
DSC	No exotherms <450°F
Nitrate	<40% by weight
Nitrite	<40% by weight
Condition 1 Hydroxide Nitrite	When $[NO_3] \leq 1.0 M$ : $0.01M \leq [OH] \leq 5.0M$ $0.011M \leq [NO_2] \leq 5.5M$
Condition 2 Hydroxide Nitrite	When $1.0M < [NO_3] < 3.0 M$ : $0.01 \times [NO_3] \leq [OH] \leq 10M$ $[OH] + [NO_2] \geq 0.4 \times [NO_3]$
Condition 3 Hydroxide Nitrite	When $3.0M < [NO_3] < 5.0 M$ : $0.3M \leq [OH] \leq 10M$ $[OH] + [NO_2] \geq 1.2$

No DSC exotherms were observed on any of the Tank 107-AP samples.

Nitrate was determined to be approximately 1019  $\mu\text{g/ml}$ , which equates to 0.016M or 0.10 percent by weight.

Hydroxide was determined to be less than 250  $\mu\text{g/ml}$  for each of the samples (except for V23 duplicate, which was 266  $\mu\text{g/ml}$ ). If it is assumed that the hydroxide concentration of all samples is the same and that the concentration is near 250  $\mu\text{g/ml}$  because V23 duplicate was slightly greater than the detection limit, then the average concentration is approximately 250  $\mu\text{g/ml}$ , which is approximately 0.015M.

Nitrite was determined to be approximately 23,480  $\mu\text{g/ml}$ , which equates to 0.489M or 2.35 percent by weight.

The nitrate molar concentration of 0.016 places condition 1 (Table 8) into effect. Under condition 1, the boundary condition for hydroxide concentration (which was determined to be approximately 0.015M in Tank 107-AP) was tentatively within limits because it was greater than the lower limit of 0.01M and less than the upper limit of 5.0M. The certainty of meeting this boundary

is questionable because most of the analytically derived values were less than the detection limit. A lower detection limit could have been achieved with a larger sample aliquot, however because of the small sample volume delivered to the laboratory, it was necessary to use small sample aliquots, conserving sufficient sample to perform all required analyses.

Under condition 1, the boundary condition for nitrite concentration (which was determined to be 0.489M in Tank 107-AP) was within limits because it was greater than the lower limit of 0.011M and less than the upper limit of 5.5M.

## QUALITY CONTROL REQUIREMENTS

### STANDARDS

The laboratory control standards (LCS) which were discussed in numerous places of the source documents (WAP, QAPJP, SOW, and TPP) were interpreted to mean Laboratory Measurement Control System (LMCS) standards, which are specific to the 222-S Laboratory. Statistical data on LMCS standards were discussed previously in the Statistical Evaluation section. These standards are generated in house by a special group within 222-S Laboratory using controlled procedures. Analyte concentrations within these standards are known to the analysts.

LMCS control limits (typically  $3\sigma$ ) existing at the time of analysis were used to meet laboratory control standard (LCS) control limit requirements, as described in the TPP, section 4.2. For some procedures (for example ICP), an administrative control limit was used rather than a statistically derived limit. In the case of ICP, not enough analytical data had been produced to generate statistically valid control limits, so limits of 85 to 115 percent were set.

Quality control status assessment of the procedure was determined using percent recovery of the nondigested LMCS standards. If an LMCS standard failed the control criteria, no data were reported from that batch.

No LMCS standard was available for the following analytes: appearance, Cs<sup>134</sup>, Ce<sup>144</sup>, Ru<sup>103</sup>, Ru<sup>106</sup>, Nb<sup>94</sup>, Eu<sup>154</sup>, Eu<sup>155</sup>, Se<sup>79</sup>, Cm<sup>244</sup>, Ra<sup>226</sup> and Pu<sup>238</sup>.

The QAPJP, section 3.1, states the following regarding acceptance criteria of LMCS standard data generated by ICP.

"When at least 10 analytes fail to meet the LCS recovery, the sample must be rerun. Only one rerun will be performed. If the rerun results are unacceptable and remain outside the QC specifications, the rerun set of results will be reported in the data package narrative as a potential quality control problem."

This statement was interpreted to mean that if 10 or more of the 12 required analytes determined by ICP fail to meet the 85 to 115 percent control limit criteria for LMCS standards, then those data will be rejected and one rerun be performed.

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LMCS control limits for each analyte are discussed in the Results of Analyses section later in the narrative.

### BLANKS

One reagent blank was analyzed with each batch of samples except for appearance and DSC. One preparation blank was analyzed with each batch of acid digested samples.

The QAPJP, section 8.2, required that the field blank be analyzed for only these ICP parameters: Ag, Al, As, Ba, Cd, Cr, Na, and Pb. During a telephone conversation in late June, 1993, between the evaporator project cognizant engineer and the 222-S Laboratory project coordinator prior to sampling, it was agreed that a single determination of the field blank for each radiochemistry analysis would be performed whenever possible. It was also agreed that duplicate analyses of the field blank would be performed on each inorganic analysis whenever possible.

A field blank was analyzed for the following constituents: Ag, Al, Ba, Ca, Cd, Cr, Fe, Mg, Mn, Na, Pb, Zn, As, Se, Hg, OH<sup>-</sup>, TIC, CN<sup>-</sup>, U, NH<sub>4</sub><sup>+</sup>, F<sup>-</sup>, Cl<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, NO<sub>2</sub><sup>-</sup>, PO<sub>4</sub><sup>-3</sup>, SO<sub>4</sub><sup>2-</sup>, Ce<sup>144</sup>, Cs<sup>134</sup>, Cs<sup>137</sup>, Co<sup>60</sup>, Eu<sup>154</sup>, Eu<sup>155</sup>, Nb<sup>94</sup>, Ra<sup>226</sup>, Ru<sup>106</sup>, total alpha, and total beta.

Blanks were considered contaminated when any constituent was determined to be equal to or greater than 20 percent of the average concentration of that analyte in the sample. If both the blank and sample were detectable but very near the detection limit, the preceding criterion did not apply.

### DUPLICATE ANALYSIS

Duplicate analyses were performed on every sample for every analyte except for appearance. No quality control criteria were specified in any of the source documents, and no reruns were performed when it appeared that a significant difference occurred between a sample and its duplicate.

### SPIKE/SPIKE DUPLICATE ANALYSIS

The QAPJP, section 8.4, 8.5, and 8.7, required spike/spike duplicate analyses on the following parameters: each of the analytes by the ICP, atomic absorption spectrophotometry and ion chromatography procedures, and for TIC, CN<sup>-</sup>, U, NH<sub>4</sub><sup>+</sup>, H<sup>3</sup>, C<sup>14</sup>, total alpha, total beta, Nb<sup>94</sup>, and Ru<sup>106</sup>. Table 15A of the QAPJP has inconsistencies compared with the narrative in its sections 8.4, 8.5 and 8.7. When inconsistencies appeared, priority was given to the narrated sections.

One spike and one spike duplicate were performed for each of the procedures above for each sampling event. Sampling event was defined (consistent with

QAPJP, section 8.2, paragraph 1) to mean all sample collections from a tank. In practice, one spike/spike duplicate was analyzed for each of the analytes by the ICP, atomic absorption spectrophotometry and ion chromatography procedures, and for TIC, CN<sup>-</sup>, U, NH<sub>4</sub><sup>+</sup>, Cs<sup>137</sup>, Co<sup>60</sup>, Am<sup>241</sup>, H<sup>3</sup>, C<sup>14</sup>, Tc<sup>99</sup>, Sr<sup>90</sup>, I<sup>129</sup>, Np<sup>237</sup>, Pu<sup>239/240</sup>, total alpha, and total beta.

It was not possible to perform a spike/spike duplicate on Nb<sup>94</sup> and Ru<sup>106</sup> because they are analyzed by gamma energy analysis, for which the only LMCS standards that exist are for Cs<sup>137</sup> and Co<sup>60</sup>. All gamma energy analyses fall into this category, except, of course, for Cs<sup>137</sup> and Co<sup>60</sup>.

For an evaluation of accuracy, the QAPJP required a recovery on all spiked samples of 75 to 125 percent, except for DSC (which required recovery limits of 90 to 110 percent). A stipulation was made in the TPP that the 75 - 125 percent recovery criteria were valid only when the analyte concentration in the spiked sample was increased by at least 25 percent more than the original sample concentration. In the case of DSC, it was not possible to measure percent recovery because the data are qualitative. That is to say, that data generated by DSC on each sample either detected exotherms or did not detect exotherms. As is noted later in the Results of Analyses section, no DSC exotherms were observed in any sample.

To evaluate precision, the QAPJP required a relative percent difference (RPD) between the spike and spike duplicate of equal to or less than 20 percent. A stipulation was made in the TPP that this criterion was valid only when the analyte concentrations of the spike and spike duplicate were greater than ten times the detection limit.

## RESULTS OF ANALYSES (DATA SUMMARY AND EVALUATION)

### PHYSICAL ANALYSES

#### APPEARANCE/HOMOGENEITY

Observations were performed on the direct (unmodified) sample. No instruments were used, consequently there was no instrument calibration. No quality control criteria were defined in the source documents nor were they required.

All analyses were performed on 8/10/93. SW-846 does not define a holding time criteria for this parameter.

Analyses were performed by procedure number LA-519-151/E-1 at 24°C and are shown in Table 9.

Table 9. Appearance/Homogeneity Analysis Summary				
Sample ID	Lab ID	Visual Observations	Dose Rate	Sample Size
V21	107-AP-A	Colorless, clear, no solids, no phases	<2mR	125 ml
V23	107-AP-B	Colorless, clear, no solids, no phases	<2mR	125 ml
V26	107-AP-C	Colorless, clear, no solids, no phases	<2mR	125 ml
V27	107-AP-D	Colorless, clear, no solids, no phases	<2mR	125 ml
V28	107-AP-E	Colorless, clear, no solids, no phases	<2mR	125 ml
V34	107-AP-COM	Colorless, clear, no solids, no phases	<2mR	125 ml

From the visual appearance, all of the samples were homogeneous. They did not require heating or dilution to maintain solubility.

#### DIFFERENTIAL SCANNING CALORIMETRY

Analyses were performed on the direct sample using procedure/revision number LA-514-113/A-0 (Differential Thermal Analysis) and instrument number WC16134.

No unusual instances or problems occurred during the analyses of DSC. SW-846 protocol do not specify a hold time for DSC. All analyses were performed on 8/31/93, 9/1/93 and 9/9/93, where holding times ranged from 30 to 39 days. The analyses of batch 1507 was split into two days with the standard, V19, being analyzed on both 8/31/93 and 9/1/93.

The statistically derived LMCS control limits for DSC are unusual. Of the nine observations in the LMCS data base (from 7/23/93 to 9/8/93), all were 100.0 percent recovery, obviously with an average recovery of 100.0 percent. This occurs because the measured analytical parameter was qualitative not quantitative. That is to say that either an exotherm was observed or it was not (100 percent or 0 percent), rather than a numerically derived value.

The QAPjP requires an LMCS percent recovery of 90 to 110 percent. The LMCS control limits for DSC are 99.9 to 100.1 percent recovery for electronic data processing (EDP) code number S230. DSC analyses of LMCS standards for tank 107-AP yielded recoveries of 100.0 percent, and met QAPjP and LMCS quality control criteria.

The DSC standard is not really a quantitative calibration standard, but is rather a matrix check sample containing a mixture of compounds which simulate the contents of single shell tanks. It was not intended to be used for evaluation of percent recovery. Indium metal is used to calibrate the instrument.

Exotherms were observed as expected in the standards of both batches, but no exotherms were observed in any of the samples. See Table 10. No blank was available for DSC.

Table 10. Summary of DSC Results	
Sample	Analytical Results
V19 (STD)	exotherm occurred between approximately 210°C and 340°C.
V19 (STD)	exotherm occurred between approximately 200°C and 340°C.
V21	no exotherms
V21 dupl	no exotherms
V23	no exotherms
V23 dupl	no exotherms
V24 (STD)	exotherm occurred between approximately 220°C and 340°C.
V26	no exotherms
V26 dupl	no exotherms
V27	no exotherms
V27 dupl	no exotherms
V28	no exotherms
V28 dupl	no exotherms

**SPECIFIC GRAVITY**

Specific gravity analyses were performed on direct samples on 8/22/93 (holding time = 21 days), using procedure/revision number LA-510-112/C-2 (reviewed on 6/26/92) and instrument number WA90787.

No unusual instances or problems occurred during the analyses of specific gravity.

For specific gravity EDP code number S332, the LMCS control limits are 96.81 to 100.53 percent recovery. An average recovery of 98.65 percent and a percent standard deviation of 0.5854 was generated from 10 new observations between 7/23/93 through 9/8/93.

For Tank 107-AP samples, LMCS standard recoveries (97.9 and 98.4 percent) were within acceptance limits. The reagent blank values were 0.979 and 0.986 (relative to an expected value of 1.000 for pure water). Spiked samples were not required nor analyzed. Precision between each sample and its duplicate

ranged from 0.0 to 4.04 relative percent difference. No Relative Percent Difference (RPD) quality control criteria were specified in the source documents between samples and their duplicates.

Specific gravity values for the samples were nearly equivalent to blank values, indicating that Tank 107-AP constituents were low in salt concentration. The grand average of sample averages was 0.989.

## INORGANIC ANALYSES

### ION CHROMATOGRAPHY

All ion chromatography (IC) analyses were performed on dilutions of the direct samples, using procedure/revision numbers LA-533-105/C-0 and LA-533-105/C-1, and instrument number WB54428. Samples V19-V23 were originally analyzed on 8/11/93 and 8/16/93 (holding times of 10 and 15 days). Samples V24 through V28 were done on 8/11/93, 8/12/93 and 8/16/93 (holding times of 10, 11, 15 days). Samples V29-V31 were analyzed on 8/12/93 (an 11 day holding time). Samples V19-V21 were rerun for fluoride only on 9/7/93 (a holding time of 37 days). Of those analytes determined by IC, only chloride, nitrate, and sulfate have holding times specified in SW-846 protocol. Those holding times for chloride, nitrate and sulfate are 28 days, 48 hours, and 28 days, respectively. Consequently, the only analyte which failed to meet the required holding time was nitrate. As was discussed above, Tank 107-AP samples were not preserved. Due, however, to the native characteristics of high pH and radioactivity, the samples would be unlikely to be subject to biodegradation, which is generally the greatest source of deterioration relative to nitrate.

The detection limit for all of the IC analytes was set at the concentration equivalent to the lowest standard within the calibration curve multiplied by 10, which was the dilution factor based on the "water leach" pretreatment. For some analytes, the *sample* detection limit was generated by multiplying the above detection limit by an additional factor based on the sample aliquot that was injected into the IC (that is to say, 11 or 101).

### FLUORIDE

For fluoride (EDP code R974), the LMCS control limits were administratively set, ranging from 90.0 to 110 percent recovery. During the period of 7/23/93 to 9/8/93 with a total of 43 new observations, the average recovery within the data base was 97.89 percent with a percent standard deviation of 3.15. LMCS recoveries for all Tank 107-AP samples were acceptable with values ranging from 95.3 percent to 97.1 percent.

A partially coeluting peak (unidentified compound) interfered with the integration of the fluoride peak. An attempt to enhance quantitation on the original V21 sample, using a manual integration with an 11-fold sample dilution to reduce peak overlap, did not appear to be effective as evidenced by the unacceptable recoveries of the spike and spike duplicate of -156.6 percent and -152.5 percent, respectively. Subsequent reruns of sample V21

still yielded unacceptable percent recoveries for the spike and spike duplicate, ranging from 60.8 to 68.0 percent.

Precision between the original V21 spike and spike duplicate was an acceptable 3.0 RPD, with rerun RPDs of 2.9 and 1.6 percent. RPDs between the samples and their duplicates ranged from 0.7 to 6.5, indicating good precision.

The reagent blanks and field blank fluoride concentrations were less than the detection limit of 1  $\mu\text{g/ml}$ .

Average fluoride concentrations of the samples ranged from <11.0 to 146  $\mu\text{g/ml}$ , with the grand average of 101  $\mu\text{g/ml}$ . These fluoride values are questionable with regard to accuracy and should be used as estimates only.

### CHLORIDE

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

For chloride (EDP code R972), the LMCS control limits were administratively set, ranging from 90.0 to 110 percent recovery. During the period of 7/23/93 to 9/8/93 with a total of 48 new observations, the average recovery within the data base was 98.70 percent with a percent standard deviation of 4.54. LMCS recoveries for Tank 107-AP samples were acceptable at 94.8 percent, 99.3 percent and 95.7 percent.

Acceptable accuracy as indicated by recoveries of the spike and spike duplicate were 89.0 percent and 88.3 percent, respectively.

Precision between the spike and spike duplicate was an acceptable 0.8 RPD. RPDs between the samples and their duplicates were undeterminable because all sample values were less than the sample detection limit of 22  $\mu\text{g/ml}$ . The sample detection limit was 11 fold greater than the instrument detection limit due to an 11 fold greater dilution.

The reagent blanks and field blank chloride concentrations were less than the detection limit of 2  $\mu\text{g/ml}$ .

### NITRATE

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte. The required sample holding time of 48 hours (a quality assurance parameter) was not met for nitrate as was discussed above.

For nitrate (EDP code R978), the LMCS control limits were administratively set, ranging from 90.0 to 110 percent recovery. During the period of 7/23/93 to 9/8/93 with a total of 43 new observations, the average recovery within the data base was 94.58 percent with a percent standard deviation of 2.36. LMCS recoveries for Tank 107-AP samples were acceptable at 93.4 percent, 97.5 percent and 94.1 percent.

Acceptable accuracy as indicated by recoveries of the spike and spike duplicate were 99.8 percent and 95.5 percent, respectively.

Precision between the spike and spike duplicate was an acceptable 0.6 RPD. RPDs between the samples and their duplicates ranged from 0.0 to 2.0, indicating good precision.

The reagent blanks and field blank nitrate concentrations were less than the detection limit of 10  $\mu\text{g}/\text{ml}$ .

Average nitrate concentrations of the samples ranged from 987 to 1,060  $\mu\text{g}/\text{ml}$ , with the grand average of 1,019  $\mu\text{g}/\text{ml}$  (0.016M).

Relative to environmental levels, Tank 107-AP nitrate concentrations are high. As a point of reference, the maximum contaminant level for nitrate in drinking water is 44  $\mu\text{g}/\text{ml}$  (as  $\text{NO}_3$ ). Relative to typical double shell tanks, however, Tank 107-AP nitrate values are quite low.

#### NITRITE

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

For nitrite (EDP code R968), the LMCS control limits were administratively set, ranging from 90.0 to 110 percent recovery. During the period of 7/23/93 to 9/8/93 with a total of 46 new observations, the average recovery within the data base was 97.11 percent with a percent standard deviation of 2.997. LMCS recoveries for Tank 107-AP samples were acceptable at 97.0, 103.4 and 102.7 percent.

Acceptable accuracy as indicated by recoveries of the spike and spike duplicate were 90.4 percent and 96.1 percent, respectively.

Precision between the spike and spike duplicate was an acceptable 1.1 RPD. RPDs between the samples and their duplicates ranged from 0.0 to 3.1, indicating good precision.

The reagent blanks and field blank nitrite concentrations were less than the detection limit of 10  $\mu\text{g}/\text{ml}$ .

Average nitrite concentrations of the samples ranged from 22,800 to 25,300  $\mu\text{g}/\text{ml}$ , with the grand average of 23,480  $\mu\text{g}/\text{ml}$  (0.489M).

Environmentally, nitrite is usually undetectable ( $<0.1 \mu\text{g}/\text{ml}$ ). Tank 107-AP nitrite concentrations are high, but relative to typical double shell tanks, the values are low.

#### PHOSPHATE (ORTHO-PHOSPHATE)

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

For phosphate (EDP code R976), the LMCS control limits were administratively set, ranging from 90.0 to 110 percent recovery. During the period of 7/23/93 to 9/8/93 with a total of 41 new observations, the average recovery within the data base was 99.82 percent with a percent standard deviation of 2.45. LMCS recoveries for Tank 107-AP samples were acceptable at 101.4, 98.5, and 101.5 percent.

Acceptable accuracy as indicated by recoveries of the spike and spike duplicate were 85.4 percent and 85.1 percent, respectively.

Precision between the spike and spike duplicate was an acceptable 0.2 RPD. RPDs between the samples and their duplicates were undeterminable because all sample phosphate values were less than the sample detection limit of 10  $\mu\text{g/ml}$ .

The reagent blanks and field blank phosphate concentrations were less than the detection limit of 10  $\mu\text{g/ml}$ .

### SULFATE

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

For sulfate (EDP code R970), the LMCS control limits were administratively set, ranging from 90.0 to 110 percent recovery. During the period of 7/23/93 to 9/8/93 with a total of 44 new observations, the average recovery within the data base was 100.53 percent with a percent standard deviation of 2.03. LMCS recoveries for Tank 107-AP samples were acceptable at 100.5 percent, 100.8 percent and 101.8 percent.

Accuracy was acceptable as indicated by a 96.3 percent recovery for both the spike and spike duplicate.

Precision between the spike and spike duplicate was an acceptable 0.0 RPD. RPDs between the samples and their duplicates was indeterminable for samples V21, V23, V27 and V28 because they were determined to be less than the detection limit of 10  $\mu\text{g/ml}$ . Sample V26 had a relative percent difference of 0.5 percent.

The reagent blanks and field blank sulfate concentrations were less than the detection limit of 10  $\mu\text{g/ml}$ .

Average sulfate concentrations of the samples ranged from <10 to 203  $\mu\text{g/ml}$ , with the grand average of 48.6  $\mu\text{g/ml}$ . For calculation purposes, "<" values were removed from the <10 values, then averaged directly.

The sulfate concentration in Tank 107-AP is within the range of many western USA drinking water sources.

### AMMONIA (by KJELDAHL and TITRATION)

Ammonia was analyzed on the direct samples using procedure/revision number LA-634-102/D-1. Instrument number AL-10696 was used for all samples.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Samples V19 through V23 were analyzed on 8/30/93, which is equivalent to a sample holding time of 29 days. Samples V24 through V28 were analyzed on 8/31/93, a sample holding time of 30 days, and samples V29 through V31 were analyzed on 8/20/93, a holding time of 19 days. Although the project coordinator could not find a citation in SW-846 protocol for sample holding time of ammonia, durations in the range of 19 to 30 days between sample collection and analysis seemed unacceptably long, especially for unpreserved samples. The accepted method for ammonia preservation is to acidify samples at the time of collection to pH <2 with nitric acid. Although biodegradation was not expected to be a significant factor in decomposition of Tank 107-AP samples due to high pH (ranging from 10.1 to 10.8) and lethal radioactivity, it was expected and quite likely that, due to the high pH of these samples, sample degradation would occur due to chemical reactions. At high pH, ionic ammonia reacts with hydroxide to generate  $\text{NH}_3$ , which is volatile and readily is dissipated at ambient temperature. Thus under the conditions of sample collection and storage prior to analyses, it is not unreasonable to believe that ammonia data are biased low.

It should be clearly understood that those chemical reactions causing changes in the sample (noted above) have been and continue to be occurring naturally in the tank. Consequently, actual tank ammonia concentration may be represented quite well by the analytical data because such processes are occurring in parallel.

Statistically derived LMCS control limits for the ammonia standard (EDP code number S235) during the period of 7/23/93 through 9/8/93 were 77.16 to 122.40 percent recovery. With 15 new observations during that period, the average recovery was 97.63 percent, and the percent standard deviation was 4.61. Actual LMCS standard recoveries for 107-AP analyses were acceptable at 97.2 percent, 93.5 percent, and 97.2 percent for the three batches.

Ammonia accuracy, as determined by evaluation of spike and spike duplicate recoveries, was not acceptable with values of 125.5 percent and 125.2 percent. Although accuracy acceptance limits ranged from 75.0 to 125.0 percent recovery, with the spikes only slightly beyond the acceptance limits, a rerun was not ordered because the sample and sample duplicate values were uniformly less than the detection limit. A rerun would likely produce sample values equivalent to those of the original run, with no improvement in the reportable values.

Precision was determined to be acceptable with a 0.0 RPD between the spike and its duplicate. Precision between samples and their duplicates was not able to be calculated because all values were "less than" values.

Ammonia concentration was less than the detection limit for all reagent blanks and for the field blank, indicating that contamination was not a problem for the ammonia analysis.

Average sample concentrations were also not able to be calculated, however all values were <40  $\mu\text{g}/\text{ml}$   $\text{NH}_3$ .

## HYDROXIDE (by TITRATION)

Hydroxide by procedure/revision number LA-211-102/B-0 was performed on the direct samples, using instrument number WCO6695, on 8/11/93.

The autotitrator pH was calibrated using standards of pH 7.00 and 10.00 with a slope of 0.971 and a voltage offset of -10.6mV. During sample analyses, all titration endpoints were within the calibration range of pH 7 to 10 (except for the blanks as expected, where the pH shift was extreme with a minute addition of HNO<sub>3</sub> titrant).

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

The sample holding time was 10 days. A required sample holding time was not specified in SW-846 protocol, however, as was stated above for ammonia, a chemical reaction of hydroxide with ammonia would be expected to cause a decrease in hydroxide concentration over time. The reversible chemical reaction equation is as follows.  $\text{NH}_4^+ + \text{OH}^- \leftrightarrow \text{NH}_3\uparrow + \text{H}_2\text{O}$

Typical titration curves were seen in the analyses of Tank 107-AP samples in contrast to Tank 101-AP samples (which was discussed in the Tank 101-AP case narrative. This was believed to be because of the absence of ammonia, which was shown to cause interference with Tank 101-AP samples using this procedure. With the absence of ammonia in the tank at present, it is not likely that hydroxide losses are presently occurring in the tank, as was the possibility stated in the preceding paragraph.

No spikes were required by the source documents, consequently, no percent recovery data are available because no spikes were analyzed.

Statistically derived LMCS control limits for the hydroxide standard (EDP code number S273) during the period of 7/23/93 through 9/8/93 were 93.28 to 108.71 percent recovery with 37 observations in the data base. During that period, the average recovery was 101.63 percent, with a percent standard deviation of 2.51. Actual LMCS standard recoveries for 107-AP analyses were acceptable at 100.2 percent, 101.5 percent and 102.4 percent for the three batches.

Precision of the sample and sample duplicate was not able to be calculated due to the "less than" sample values.

Reagent blanks are used to correct sample values, consequently it is not possible to "analyze" a reagent blank since a blank would be subtracted from the blank to yield a result of zero. Field blank hydroxide concentrations were less than their detection limits, which were dependent on the determination's aliquot size.

Average hydroxide concentrations were not able to be calculated because of the "less than" sample values, however the estimated hydroxide concentration of all samples was <250 µg/ml.

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**CYANIDE (by DISTILLATION/SPECTROPHOTOMETRY)**

Cyanide analyses were performed on water diluted samples using procedure/revision number LA-695-102/B-0, and instrument number AL10724. These analyses were done on samples V19 through V28 on 8/30/93, and on samples V29 through V31 on 8/31/93, with sample holding times of 29 days and 30 days, respectively. A maximum sample holding time was not specified for this analyte in SW-846 protocol.

For this procedure, full calibration curves were not generated at the time of analysis. A standard curve was generated on November 11, 1992, where the following curve statistics were obtained: Y intercept = 0.000141, slope = 0.1718, and the correlation coefficient ( $r^2$ ) = 0.99998. These curve fitting parameters were applied to evaporator samples V19 through V31. For Tank 107-AP cyanide analyses, the LMCS standard had additional importance because it was used to check the validity of the standard curve which was generated several months prior.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

The cyanide limit of detection was 0.1  $\mu\text{g}$ . Aliquot volumes for samples and blanks V20 through V23 were 4.00 ml, and were 5.00 for V25 through V28, V30 and V31. By calculation, the detection limit for V20 through V23 was 0.25  $\mu\text{g}/\text{ml}$  and for V25 through V28, V30 and V31 was 0.20  $\mu\text{g}/\text{ml}$ . The concentrations of reagent blanks and field blanks were determined to be less than their detection limits.

Accuracy of the spike and spike duplicate determinations were within acceptance limits with percent recoveries of 94.7 and 95.0.

Statistically derived LMCS control limits for the cyanide standard (EDP code number S244) during the period of 7/23/93 through 9/8/93 were 89.24 to 104.92 percent recovery. Thirteen new observations during that period yielded an average recovery was 96.81 percent, with a percent standard deviation of 1.97. Accuracy of LMCS standard determinations was acceptable with percent recoveries of 94.5, 96.4, and 95.8.

Precision between the spike and spike duplicate was acceptable with 0.3 RPD.

Only sample V26 had a large RPD (22.2 percent) between the sample and its duplicate. The other samples had small RPDs ranging from 0.0 to 7.7 percent.

Average sample values were approximately 27 fold greater than the detection limit. Average sample values ranged from 0.52 to 0.65  $\mu\text{g}/\text{ml}$  with a grand average of 0.58  $\mu\text{g}/\text{ml}$ . Tank 107-AP appeared to be relatively homogeneous between sample points relative to cyanide concentrations.

**TOTAL INORGANIC CARBON (CARBONATE)**

Total inorganic carbon (TIC) analyses using instrument number W839937 on direct samples with procedure/revision number LA-622-102/B-2, were originally performed on all samples on 8/10/93, a holding time of 9 days. Reruns were

performed on samples V24 through V31 on 8/14/93 (a 13 day holding time), and on samples V19 through V23 on 8/20/93 (a 19 day holding time). A maximum sample holding time for this analyte was not specified in SW-846 protocol.

Statistically derived LMCS control limits for the carbonate standard (EDP code number S223) during the period of 7/23/93 through 9/8/93 were 86.93 to 108.01 percent recovery. Forty six new observations during that period yielded an average recovery of 103.33 percent and a percent standard deviation of 3.78. Accuracy of LMCS standard determinations was acceptable for the original analyses with recoveries of 99.1, 90.6 and 94.8 percent. Accuracy for rerun LMCS standards was also acceptable with recoveries of 97.0, 104.5 and 107.4 percent.

Accuracy for the spike and spike duplicate was not acceptable for the original run with percent recoveries of 62.1 and 58.9, respectively. As a consequence a rerun was performed. The rerun spike and spike duplicate recoveries were acceptable at 100.6 and 100.8 percent respectively. The batch summary sheet narrative written by the chemist noted that the poor recovery of the *original* run's spikes was caused by incorrect instrument setup. Original data are shown on the data summary table, however the accuracy of these values is questionable.

Precision between the spike and spike duplicate was acceptable for both the original run and the rerun with RPDs of 5.2 and 0.2 respectively. Precision between samples and their duplicates was reasonable but less precise for the original run (ranging from 0.8 to 19.3 RPD) than for the rerun, which was much better with RPDs ranging from 0.0 to 0.8 percent.

The field blank's carbonate concentration was less than the detection limit of 5  $\mu\text{g}/\text{ml}$ .

In calculating the sample carbonate concentrations, an instrument blank value is subtracted from the sample value. Values shown on the data summary sheet for each reagent blank are uncorrected. For the analysis of a reagent blank, however, the result would typically be subtracted from itself, yielding a corrected blank value of zero. The detection limit shown on the data summary sheet was based on a standard volume of 0.20 milliliters (an optimal value). Generally the least amount of inorganic carbon detectable was 1  $\mu\text{g}$ . Thus, one microgram of carbon divided by 0.20 ml equaled 5.00  $\mu\text{g C}/\text{ml}$ , which was the detection limit shown for this procedure and represented the optimum aliquot. To generate a detection limit with an aliquot equivalent to that of the standard (0.05 ml), the stated detection limit must be multiplied by 4, yielding a value of 20  $\mu\text{g}/\text{ml}$ .

Average carbonate concentration of the samples (based on rerun data only) ranged from 232 to 302  $\mu\text{g}/\text{ml}$ . The grand average concentration of all samples was 270  $\mu\text{g}/\text{ml}$ .

#### TOTAL URANIUM (by LASER FLUORIMETRY)

Chemical (not radiochemical) analyses for total uranium were performed on direct samples. These determinations were performed using instrument number WB88807 by procedure/revision number LA-925-106/B-0.

Samples V19 through V23 were originally tested on 9/13/93 (a sample holding time of 43 days). Samples V24 through V28 were done on 9/22/93 (a sample holding time of 52 days). Samples V29 through V31 were tested on 9/9/93 (a sample holding time of 39 days). The first rerun of samples V19 through V23 was performed on 10/27/93 (a sample holding time of 87 days). The second rerun of samples V19 through V23 was done on 11/1/93 (a sample holding time of 92 days). A maximum sample holding time was not specified for this analyte in SW-846 protocol.

LMCS standard recovery control limits for uranium (EDP code number S267) were 77.19 and 121.70 percent. During the time period 9/9/93 through 11/2/93, when Tank 107-AP samples were analyzed, a statistical evaluation was made with 45 new observations where the average percent recovery was 100.78 and the percent standard deviation was 5.57.

LMCS standard accuracy was acceptable for Tank 107-AP samples with percent recoveries for the original analyses of samples V19 through V31 of 105.7, 100.5, and 100.4. Reruns for samples V19 through V23 generated acceptable percent recoveries of LMCS standards of 117.6 and 100.5. Note that in the data summary spreadsheets, standard results are in g/L of uranium.

Two reruns were ordered for samples V19 through V23, because of unacceptable spike and spike duplicate recoveries each of 73.3 percent recovery. It appears that an interference was observed, which was similar to that seen with Tank 101-AP samples, and was attributed to the basic pH (approximately 10.5) of the samples. An unsuccessful attempt was made to deal with the interference by taking a very small aliquot for analysis via serial dilutions. As a consequence, large errors were generated in percent recovery calculations when experimental error was multiplied by large dilution factors. This was particularly the case for the instrument used in this procedure, which is quite insensitive. For example, when a rerun of sample V21 was performed, the spike and spike duplicate were generated identically, and all numbers used in the calculation of percent recovery were identical except for the numerical value for the instrument meter reading of each determination (for Sample + Fluran + Spike). For the spike, the reading was 0.34, yielding a percent recovery of 110.8. This is contrasted with the spike duplicate, which had a reading of 0.35 (only a one tenth difference, the smallest discernable increment), yielding a percent recovery of 105.4. With this condition, it is not unlikely that large differences in recovery are due to small experimental error.

Both reruns yielded acceptable spike/duplicate-spike recoveries of 103.5 and 96.2 percent respectively for the rerun performed on 11/1/93, and of 110.8 and 105.4 percent respectively for the rerun done on 10/27/93. Both of these reruns had an acceptable spike RPD of 7.5 percent and 5.0 percent respectively.

Reagent blank values were uniformly less than the detection limit, and the field blank's concentration was only four fold greater than the detection limit. The field blank was determined to not be contaminated because its average concentration was less than 20 percent of the sample's grand average concentration.

Precision between samples and duplicates from the original sets of analyses ranged from 0.0 to 26.8 RPD with an average RPD of 12.4, with only sample V21's precision greater than 20 percent. Sample precision in the reruns of V21 was significantly better with RPDs of 6.3 and 6.0 percent, however sample V23 showed greater imprecision with RPDs of 18.1 and 66.1 percent. Precision error of this magnitude was insignificant because the sample values were generally less than five fold the detection limit. From this perspective, it appears that differences in uranium concentration between samples were insignificant, again indicating the homogeneity between samples. The average sample values ranged from 0.0138  $\mu\text{g/ml}$  to 0.0338  $\mu\text{g/ml}$ , with a grand average of 0.0234  $\mu\text{g/ml}$  of uranium.

#### ARSENIC (by HYDRIDE ATOMIC ABSORPTION SPECTROPHOTOMETRY)

Samples were acid digested prior to arsenic analyses to ensure complete solubility and better recovery. Original analyses were done on samples V19 through V28 on 9/1/93; samples V29 through V31 were tested on 9/2/93, then samples V19 through V23 were retested on 11/4/93 and 11/9/93. The holding times for these analytical runs were 31 days, 32 days, 95 days and 100 days, respectively. SW-846 protocol require a sample holding time of six months or less for arsenic. All Tank 107-AP samples were analyzed within the SW-846 required holding time.

Arsenic analyses were performed using procedure/revision number LA-355-131/B-1 and instrument numbers PE2280 (hydride AA) and WB27979. Instrument calibration data are as follows:

<u>Samples</u>	<u>Batch</u>	<u>Y-intercept</u>	<u>Slope</u>	<u>r<sup>2</sup></u>
V19-V23	1513	0.0052	0.0225	0.9994
V24-V28	1528	0.0137	0.0203	0.9973
V29-V31	1542	-0.0149	0.0219	0.9999
V19-V23	2022	-0.0160	0.0178	0.9998
V19-V23	2123	-0.0101	0.0208	0.9992

LMCS standard (EDP code R741) control limits for the period of 7/23/93 to 9/9/93 were 76.35 to 132.94 percent recovery. During that period 8 additional observations were collected where the average percent recovery was 98.75 percent and the percent standard deviation was 9.71. Undigested LMCS standards analyzed with 107-AP samples yielded acceptable accuracy with percent recoveries of 102.2, 103.9 and 105.2 for the original analytical runs. Percent recoveries of digested LMCS standards for those runs were significantly higher with percent recoveries of 139.0, 129.1 and 134.2, indicating the possibility of some contamination during the digestion process. For the two rerun batches, undigested LMCS standard recoveries were acceptable at 92.4 and 90.2 percent. The digested LMCS standards had recoveries that were slightly greater at 125.5 and 108.0 percent.

Accuracy of the original spike and spike duplicate were not acceptable with percent recoveries of 134.1 and 146.4, respectively. One of the rerun batches also yielded unacceptable accuracy with percent recoveries of 140.5 and 143.0 for the spike and spike duplicate, respectively. The second rerun's accuracy was acceptable with recoveries of 112.3 and 123.1 percent, respectively. The chemist's narrative for each batch prior to the last rerun commented that the

poor recoveries were possibly due to differences in the matrix between the digested and non-digested determinations. For the last rerun, the calibration standards were digested in the same matrix as the samples, resulting in significantly improved spike and spike duplicate recoveries, which reinforced the opinion stated in the prior narratives. The effect of this finding was to make clear that if there was any bias of sample values, that the bias would be high (that is to say that the "true" sample values would actually be lower than that stated). The final consequence of this was meaningless, because the samples were already uniformly less than the detection limit and could not be expressed at values less than those already expressed.

Precision was uniformly acceptable between the spike and spike duplicate for all runs with an RPDs of 8.8, 1.8 and 9.2 percent. It was not possible to determine precision between samples and their duplicates because all of the samples were less than the detection limit of 0.0130 µg/ml.

Because all of the sample arsenic concentrations were below detection limits, any positive bias due to potential contamination during the sample digestion was inconsequential.

The field blank and reagent blanks were uniformly below the detection limit.

**SELENIUM (by HYDRIDE ATOMIC ABSORPTION SPECTROPHOTOMETRY)**

Samples were acid digested prior to selenium analyses to ensure complete solubility and better recovery. Analyses were done on samples V19 through V23 on 9/7/93; samples V24 through V28 were tested on 8/24/93 and samples V29 through V31 were done on 9/8/93. The holding times for these three batches were 37 days, 23 days and 38 days, respectively. SW-846 protocol require a sample holding time of six months or less for selenium. All Tank 107-AP samples were analyzed within the required holding time.

Selenium analyses were performed using procedure/revision number LA-365-131/B-2 and instrument numbers PE2280(Hydride) and WB27979. Instrument calibration data are as follows:

<u>Samples</u>	<u>Batch</u>	<u>Y-intercept</u>	<u>Slope</u>	<u>r<sup>2</sup></u>
V19-V23	1514	0.0002	0.0096	0.9990
V24-V28	1529	-0.0036	0.0149	0.9999
V29-V31	1543	-0.0019	0.0096	0.9990

LMCS standard (EDP code R743) control limits for the period of 7/23/93 to 9/9/93 were 59.48 to 140.47 percent recovery. During that period 10 additional observations were collected where the average percent recovery was 99.00 percent and the percent standard deviation was 13.43. Undigested LMCS standards analyzed with 107-AP samples yielded acceptable accuracy with percent recoveries of 93.4, 100.6, and 96.1. Percent recoveries of digested LMCS standards were higher with percent recoveries of 116.3, 123.4 and 126.4.

Accuracy of spike and spike duplicates were acceptable with percent recoveries of 107.7 and 111.9, respectively.

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Precision was acceptable between the spike and spike duplicate with an RPD of 3.8 percent. It was not possible to determine precision between samples and their duplicates because sample selenium concentrations were determined to be less than the detection limit of 0.0130  $\mu\text{g/ml}$ .

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Because the sample concentrations were below detection limits, any positive bias due to sample digestion was believed to be inconsequential.

The field blank and reagent blanks were uniformly below the detection limit.

#### MERCURY (by COLD VAPOR ATOMIC ABSORPTION SPECTROPHOTOMETRY)

Although mercury analyses are noted on the batch sheets as being done on the direct sample (because no preparative work was performed by the Sample Preparations Group), procedure/revision number LA-325-104/A-1, a CLP derived procedure, includes a substantial digestion phase. The instruments used were PE2380 (Hydride AA) and WB26847. All analyses were performed on 9/5/93 and 9/6/93 which were holding times of 35 and 36 days. SW-846 protocol require a maximum holding time of 38 days, consequently all mercury analyses were performed within the required holding time.

<u>Samples</u>	<u>Batch</u>	<u>Y-intercept</u>	<u>Slope</u>	<u>r<sup>2</sup></u>
V19-V23	1510	0.0003	0.0105	0.9991
V24-V28	1520	-0.0001	0.0105	0.9998
V29-V31	1532	0.0041	0.0106	0.9998

The correlation coefficient for each batch was acceptable with values greater than the minimum procedure required value of 0.995.

Statistically derived LMCS control limits for the mercury standard (EDP code number R716) during the period of 7/23/93 through 9/8/93 were 76.57 to 121.40 percent recovery. During that period, with 37 new observations, the average recovery was 97.65 percent, with a percent standard deviation of 8.16. Actual LMCS standard recoveries for 107-AP analyses were acceptable at 100.9 percent, 100.1 and 98.6 percent for the three batches.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Precision between the spike and spike duplicate was an acceptable 4.9 relative percent difference. Precision between samples and their duplicates was not measurable because all sample values were less than the detection limit of 0.003  $\mu\text{g/ml}$ .

Accuracy of the spike and duplicate spike was acceptable with percent recoveries of 99.9 and 95.2.

The field blank mercury concentration was determined to be less than the sample detection limit of 0.003  $\mu\text{g/ml}$ . All reagent blanks were less than 0.005  $\mu\text{g/ml}$ .

#### INDUCTIVELY COUPLED PLASMA/EMISSION SPECTROSCOPY (ICP)

An acid predigestion was performed on ICP samples prior to analysis by procedure/revision number LA-505-151/D-0 using instrument number WB39939.

Samples V19 through V23 were analyzed initially on 9/8/93 (a 38 day holding time). Sample V21 was chosen as the representative sample upon which the quality control analyses would be performed. When preparing the spike and spike duplicate, the spiking standard did not contain calcium, magnesium or sodium. Consequently, a QC rerun was scheduled for V21. By the time that the rerun was scheduled, however, sample V21 had been completely consumed and V23 was used to generate the necessary QC parameters. That run occurred on 11/29/93 (a 120 day holding time). Samples V24 through V28 were analyzed on 9/9/93 (a 39 day holding time). Samples V29 through V31 were analyzed on 9/10/93 (a holding time of 40 days).

SW-846 protocol require that the holding time for these metals may not exceed six months. All ICP analyses were completed within the required holding time.

ICP data were reported using a CLP deliverable package produced by Ward™ Scientific, Ltd. The report generated by this software was referred to as the "Wards™ package".

The Wards™ package uses the concept of a sample delivery group (SDG), which includes samples, duplicates, spikes, blanks, instrument control standards and blanks, interference checks, serial dilutions and narrative information within a batch. SDGs are coded alphanumerically to provide additional information: an "A" suffix denotes an acid digestion, and an "R" behind other letters indicates a rerun. The designations for initial calibration verification and continuing calibration verification are ICV and CCV, respectively. These are undigested LMCS control standards. The acronyms, ICB and CCB, denote initial calibration blank and continuing calibration blank (undigested reagent blanks).

Undigested blanks were used to initially calibrate the ICP instrument and to check calibration on a continuing basis. These blanks are referred to as ICB and CCB, respectively. A digested reagent blank included with each batch, however, was used to determine the extent of blank contamination introduced during sample preparation (and by inference, the estimated amount of sample contamination).

As was specified in the TPP, section 4.2, accuracy evaluation criteria for ICP LMCS standards was based on undigested initial calibration verification (ICV) standards. LMCS control limits were set administratively for all metals at 85.00 to 115.00 percent recovery during the period in which tank 107-AP samples were analyzed. This was because the newly developed LMCS standard, ICPST4, was first used on July 1, 1993, and there had been an insufficient quantity of data to establish statistically derived control limits. During

the period of 7/23/93 through 10/15/93, additional LMCS data were collected on ICPST4 and are summarized in Table 11.

Table 11. New ICP LMCS Data				
Analyte	EDP Code	# Observations	Average % Rec	% Std Deviation
Ag	S157	48	99.14	2.55
Al	S101	54	97.78	3.31
Ba	S107	51	100.58	2.09
Ca	S117	52	100.99	3.71
Cd	S115	49	100.04	2.65
Cr	S121	54	101.17	2.54
Fe	S129	51	99.78	1.77
Mg	S137	50	98.49	2.06
Mn	S139	50	99.72	2.07
Na	S159	52	100.21	2.15
Pb	S133	49	99.69	2.70
Zn	S179	36	99.10	1.85

In the Wards™ package, if the reported values were less than the detection limit, precision for duplicate analyses was reported as 200 percent RPD and as 100 percent for serial dilution RPDs.

The ICP was designed to perform interelement corrections for aluminum, calcium, iron, and magnesium. An initial screening test of the samples revealed negligible quantities of uranium, consequently no interference from uranium was expected.

Because each ICP determination generated analytical values for 12 metals simultaneously, generally there were failures within each run. Those failures were discussed in the Wards™ package narratives. In the package, form VII-IN misleadingly identifies the digested LMCS control standard as the "Laboratory Control Sample", however this package information is not able to be edited. Similarly, a footer incorrectly identifies the control limits for the laboratory control sample as 90 to 110 percent recovery.

It is instructive to note that the units reported for ICP are  $\mu\text{g/L}$  (or parts per billion), which is 1000 fold lower than the units of  $\mu\text{g/ml}$ , which are used elsewhere in this data package.

In the tabular data summaries, a column entitled, "Det. Lim., IDL,  $\mu\text{G/L}$ ", is included. This column indicates the instrument detection limit, which is the lowest detectable concentration by ICP without a sample dilution factor being applied. For several analytes, "less than" values are given for samples. These "less than" values represent the *sample's* detection limit which is equal to the instrument detection limit multiplied by the sample dilution factor. This will cause the sample detection limit to be greater than the instrument detection limit.

The linear concentration range for ICP analytes was determined on September 29, 1993. The maximum concentration within the linear range (defined as the highest concentration in which the percent recovery of a standard deviates less than five percent from 100 percent) is listed as follows:

<u>Analyte</u>	<u>Concentration (<math>\mu\text{g/L}</math>)</u>
Aluminum	300000.0
Barium	10000.0
Cadmium	10000.0
Calcium	10000.0
Chromium	300000.0
Iron	50000.0
Lead	10000.0
Magnesium	10000.0
Manganese	10000.0
Silver	50000.0
Sodium	1000000.0
Zinc	100000.0

### ALUMINUM

Percent recovery of all undigested LMCS standards (which ranged from 92.6 to 97.9) were within control limits.

All digested laboratory control samples had percent recoveries which were within the LMCS undigested standard control limits.

Accuracy was acceptable as indicated by sample V21 spike and spike duplicate recoveries of 97.4 percent and 98.5 percent, respectively. For the QC rerun, sample V23 spike and spike duplicate percent recoveries were 104.3 and 103.8, respectively.

Precision between sample V21 spike and spike duplicate was an acceptable 1.1 relative percent difference. Precision between sample V23 spike and spike duplicate was also acceptable with a 0.4 relative percent difference.

RPDs between the samples and their duplicates ranged from 6.9 to 40.4 percent. Only tank samples V27 and V28, and the field blank had RPDs greater than 20 percent, which was likely due to contamination during the digestion, or due to the generally low aluminum concentration overall as the chemist's narrative suggested.

The instrument detection limit was determined to be 22  $\mu\text{g/L}$ , however because the sample was diluted five fold during digestion, the sample detection limit was 110  $\mu\text{g/L}$ . The average sample aluminum concentrations were approximately 10 fold greater than the sample detection limit. Because the sample concentrations were significantly greater than the detection limit, the criterion was effective which determines when a blank is contaminated. The preparation blank for V23 rerun was determined to be contaminated because it exceeded 20 percent of the average sample concentration. The case narrative generated by the cognizant chemist erroneously stated that the preparation blank for samples V26, V27 and V28 was contaminated. Although aluminum was detected at approximately 15 percent in the preparation blank, it was less than the 20 percent contamination criterion.

The field blank's aluminum concentration was only slightly less (74 percent) than that of the samples. This indicates that contamination occurred during

processing of the sample and raises the possibility that such contamination was possible within the samples. The records indicate, however, that the acid digestions were performed on different days for each of these analytical batches.

Average aluminum concentrations of the samples ranged from 864 to 1420  $\mu\text{g/L}$ , with a grand average of 1083  $\mu\text{g/L}$ . With consideration for the greater amount of imprecision expected at part per billion levels, the aluminum concentration of each of the samples appears to be quite similar.

### BARIUM

Percent recovery of all undigested LMCS standards (which ranged from 98.1 to 102.5) were within control limits.

All digested laboratory control sample percent recoveries were within the range of 90.0 to 98.6 percent, and were within the LMCS undigested standard control limits.

Accuracy was acceptable as indicated by sample V21 spike and spike duplicate recoveries of 98.9 percent and 99.5 percent, respectively. For the QC rerun, sample V23 spike and spike duplicate percent recoveries were 97.5 and 100.1, respectively.

Precision between sample V21 spike and spike duplicate was an acceptable 0.6 relative percent difference. Precision between sample V23 spike and spike duplicate was also acceptable with a 2.7 relative percent difference.

RPDs between the samples and their duplicates ranged between 10.6 and 30.0 percent. Only tank sample V28 had an RPD greater than 20 percent, which was likely due to contamination during the digestion, or due to the generally low barium concentration (approximately two fold greater than the detection limit) as the chemist's narrative suggested.

Field blank and reagent blank concentrations were less than the detection limit.

Average barium concentrations of the samples ranged from <15 to 44.6  $\mu\text{g/L}$ , with a grand average less than or equal to 23.9  $\mu\text{g/L}$  (less than values were deleted to perform the calculations).

### CADMIUM

The percent recoveries of all undigested LMCS standards (which ranged from 97.4 to 102.4) were within control limits.

All digested laboratory control sample percent recoveries were within the range of 86.2 to 97.2 percent, and were within the LMCS undigested standard control limits.

Accuracy was acceptable as indicated by sample V21 spike and spike duplicate recoveries of 98.6 percent and 98.1 percent, respectively. For the QC rerun, sample V23 spike and spike duplicate percent recoveries were also acceptable at 93.1 and 96.8, respectively.

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Precision between sample V21 spike and spike duplicate was an acceptable 0.5 relative percent difference. Precision between sample V23 spike and spike duplicate was also acceptable with a 3.9 relative percent difference.

RPDs between the samples and their duplicates were not able to be calculated because all sample values were less than the detection limit.

Field blank and reagent blank concentrations were less than the detection limit.

The average cadmium concentrations of the samples was  $<20 \mu\text{g/L}$ .

### CALCIUM

Percent recovery of all undigested LMCS standards (which ranged from 101.5 to 105.3) were within control limits.

Digested laboratory control samples which were included with the field blank, V31, and with the V23 rerun had percent recoveries of 111.5 and 119.0 percent, respectively, and were within the LMCS undigested standard control limits. Those digested control samples which were included with the V19-V23 run and with the V24-V28 run had percent recoveries of 127.8 and 139.3 percent, respectively. Although these samples had no required QC limits, it is certain that there was a significant problem with the quality of the data, which suggests that contamination of these LCS samples occurred during digestion. Additional evidence of the contamination of samples during digestion was observable in the preparation blanks. Those blanks for samples V21, V26 and V27 were determined to be contaminated by having a calcium concentration greater than 20 percent of the sample. All digested preparation blank concentrations ranged from 75 to 108 fold greater than the sample detection limit. Conversely, undigested reagent blanks were determined uniformly to have calcium concentrations which were less than the detection limit.

The average field blank concentration was 2352 fold greater than the sample detection limit.

Accuracy appeared to be acceptable as indicated by sample V23 spike and spike duplicate recoveries of 93.6 percent and 89.5 percent, respectively.

Precision between sample V23 spike and spike duplicate was acceptable with a 2.8 relative percent difference.

RPDs between the samples and their duplicates ranged between 15.3 and 192.5 percent. Only tank sample V27 had an RPD less than 20 percent. The poor precision also pointed to contamination during the digestion.

Average calcium concentrations of the samples ranged from 2240 to 61,500  $\mu\text{g/L}$ , with a grand average (estimate) of 26,573  $\mu\text{g/L}$ . Although these five Tank 107-AP samples are relatively equivalent, that is to say that the tank contents appear to be homogeneous with respect to nearly all of the constituents, there was a 27 fold difference between the sample with the lowest average calcium concentration and that of the highest. Even the difference between the original analysis and the rerun for V23 was 26 fold for the average sample concentrations.

It is clear that the values reported for calcium on the summary spread sheet are estimates of the maximum calcium concentration in tank 107-AP samples.

Historically it has been observed that calcium contamination could be detected in digested ICP samples when plastic gloves used in the sample preparation area were not washed free of talcum powder.

### CHROMIUM

Accuracy was acceptable as indicated by recoveries of the spike and spike duplicate which were 100.3 and 100.3 percent, respectively.

Percent recovery of all undigested LMCS standards were within control limits. Those recoveries ranged from 98.8 to 103.7 percent. Although no control criteria were set for the digested laboratory control samples, these were good with recoveries ranging from 90.5 and 99.9 percent.

Precision between the spike and spike duplicate was an acceptable 0.0 relative percent difference. RPDs between the samples and their duplicates were not able to be calculated because the sample concentrations were less than the sample detection limit of 30.0  $\mu\text{g/L}$ .

The field blank and all sample preparation blank concentrations were less than the detection limit.

Sample chromium concentrations ranged from <30 to 77.6  $\mu\text{g/L}$ , with a grand average equal to or less than 38.2  $\mu\text{g/L}$  (less than values were deleted for calculations).

### IRON

Acceptable accuracy was indicated by recoveries of sample V21's spike and spike duplicate which were 98.6 and 97.8 percent, respectively. The spiking accuracy was also acceptable for V23 spike and spike duplicate, which were 101.4 and 99.0 percent, respectively.

Percent recovery of all undigested LMCS standards were within control limits, ranging from 98.6 to 101.2 percent. There were no QC criteria for digested laboratory control samples, yet these also met LMCS control limits ranging from 93.8 to 112.7 percent recovery, however the dispersion was greater.

Precision between the spike and spike duplicate was acceptable with an RPD of 0.8 percent for V21 and 2.3 percent for V23. RPDs between the samples and their duplicates ranged from not determinable to 102.5 percent. Although these five Tank 107-AP samples are relatively equivalent, that is to say that the tank contents appear to be homogeneous with respect to nearly all of the constituents, there was nearly a 10 fold difference between the sample with the lowest average iron concentration and that of the highest.

The field blank, V31, also had the highest RPD, which was likely due to greater contamination of the duplicate during the digestion than of the sample. V31's average iron concentration was greater than the highest average sample concentration, a clear indication of sample contamination.

Digested preparation blank concentrations were extremely divergent. The blank analyzed with samples V26-V28 had a reported iron concentration of "-35.7"  $\mu\text{g/L}$ , yet the blank analyzed with samples V21 and V23 had a concentration of 475  $\mu\text{g/L}$  (43 fold greater than the sample detection limit of 11  $\mu\text{g/L}$ ). These

blank failures were due potentially to contamination during sample digestion and/or to the expected variability seen at the low parts per billion levels. Because iron is easily recovered from the digestate (that is to say that it is quite soluble in the digestion matrix), it is unlikely that poor precision was caused by variable recovery from the digestion procedure.

Average iron concentrations of the samples ranged from <55.0 to 522  $\mu\text{g/L}$ , with an estimated (because of probable contamination) grand average equal to or less than 219  $\mu\text{g/L}$  (less than values were deleted to perform the calculations).

### LEAD

Acceptable accuracy was obtained for the V21 spike and spike duplicate with 108.4 and 103.9 percent recovery, respectively. Accuracy was also acceptable for the V23 spike and spike duplicate with recoveries of 95.9 and 96.4 percent, respectively.

Percent recovery of all undigested LMCS standards were within control limits, ranging from 98.1 to 101.8 percent recovery. The digested laboratory control samples had recoveries ranging from 87.8 to 100.2 percent (also within the LMCS control limit range).

Precision between the V21 and V23 spikes and spike duplicates were acceptable with relative percent differences of 4.2 and 0.5, respectively. RPDs between the samples and their duplicates were not able to be calculated (except for V28 with a 20.0 percent RPD) because one or both of the determinations from each sample was less than the sample detection limit of 190  $\mu\text{g/L}$ . Because the lead concentration of V28 was only slightly greater than the other samples (all of which were less than the detection limit) it is not likely that this sample was contaminated.

All preparation blanks had lead concentrations that were less than the detection limit of 38.0  $\mu\text{g/L}$ . Field blank values were less than its detection limit of 190  $\mu\text{g/L}$ , which, because of its dilution factor of 5, was five fold greater than the instrument detection limit.

Average sample lead concentrations ranged from <190 to 223  $\mu\text{g/L}$ , with a grand average equal to or less than 261  $\mu\text{g/L}$  (less than values were deleted for calculations).

### MAGNESIUM

Percent recovery of all undigested LMCS standards were within control limits, ranging from 95.5 to 100.0 percent. The digested laboratory control samples also met the LMCS control limit standards for the batches containing V26-V28, V31 and the V23 rerun with values ranging from 91.0 to 107.8 percent recovery. The batch containing V21 and V23 had a recovery of 179.6 percent, indicating the possibility of its contamination during digestion.

Additional evidence of the contamination of samples during digestion was observable in the preparation blanks. Those blanks for samples V26 and V23 rerun were determined to be contaminated by having a magnesium concentration greater than 20 percent of the sample. All digested preparation blank concentrations ranged from 13 to 94 fold greater than the detection limit.

Conversely, undigested reagent blanks were determined uniformly to have calcium concentrations which were less than the detection limit.

The average field blank concentration of 2,020  $\mu\text{g/L}$  was 135 fold greater than the sample detection limit. The field blank was determined to be contaminated, having an average concentration which was approximately 1.4 fold greater than the average magnesium concentration of all of the samples.

Acceptable accuracy was obtained for the V23 spike with 93.8 percent recovery and for the spike duplicate with 96.3 percent recovery.

Precision between the spike and spike duplicate was acceptable with an RPD of 2.5 percent. RPDs between the samples and their duplicates ranged between 4.5 and 163.8 percent. The poor precision also pointed to contamination during the digestion.

There was a 16 fold difference between average concentrations of the original V23 and its rerun, which casts doubt on the accuracy of magnesium. Average sample magnesium concentrations ranged from 103 to 3800  $\mu\text{g/L}$ , with a grand average of 1484  $\mu\text{g/L}$ . These data should be considered to be estimates.

### MANGANESE

Percent recovery of all undigested LMCS standards were within control limits, ranging from 97.9 to 101.2. Digested laboratory control samples yielded recoveries ranging from 89.0 to 101.1 percent, which were also within the LMCS control limits.

Acceptable accuracy was obtained for the V21 spike with 98.2 percent recovery and for its spike duplicate with 100.3 percent recovery. Accuracy was also acceptable for the V23 Spike and spike duplicate with 95.4 and 98.5 percent recovery, respectively.

Precision between the spike and spike duplicate for samples V21 and V23 were acceptable with relative percent differences of 2.2 and 3.2. RPDs between the samples and their duplicates were not able to be calculated (except for V23) because one or both of the determinations from each sample was less than the sample detection limit of 15.0  $\mu\text{g/L}$ . For sample V23, the sample/duplicate precision was 16.6 RPD.

Manganese concentrations for the field blank and its duplicate were less than the detection limit of 15.0  $\mu\text{g/L}$ . Although the preparation blanks for two of the batches had detectable manganese concentrations, those values were only slightly greater than the detection limit (as were also the sample values), and were determined to not be contaminated.

The average manganese concentration for all tank samples, except for V23, was less than 15.0  $\mu\text{g/L}$ . The average concentration for the original V23 analysis was 69.2  $\mu\text{g/L}$ , whereas the rerun was less than 15.0  $\mu\text{g/L}$ . The grand average for all samples was equal to or less than 40.3  $\mu\text{g/L}$  (less than values were deleted for calculations).

### SILVER

All LMCS standard percent recoveries were within acceptable accuracy control limits, ranging from 97.9 to 101.2.

The digested laboratory control samples, however, suggested that there may be a potential problem with the digestion. Such a digestion problem was seen in the samples from Tank 101-AP. Tank 107-AP samples were prepared by the same procedure. The digestion procedure (LA-505-158), through which all ICP samples were processed, includes a step where samples were exposed to hydrochloric acid. Because silver precipitates in a matrix with high chloride concentration, it was expected that silver recovery during analysis would be negatively impacted. For the V26-V28 and V31 batches, the digested standards yielded low recoveries of 58.3 and 54.4 percent, respectively. It was also noted however, that significant variability occurred during the analyses of these two batches, as well as for the V23 rerun batch between successive determinations of the initial calibration blank and the continuing calibration blanks. This tends to indicate that the low recoveries of digested standards may be more related to instrumental drift than to digestion problems.

Acceptable accuracy was obtained for the spike and the spike duplicate for V21 with percent recoveries of 103.3 and 102.2 percent. Spike/spike-duplicate recoveries for V23 were also acceptable with 99.2 and 98.0, respectively. The accuracy of these data, which were placed in question due to poor recoveries of two of the four digested standards, appear to be acceptable, because these spikes were digested and yet yielded acceptable recoveries.

Precision between the V21 spike and spike duplicate was acceptable with a 1.1 relative percent difference. Sample V23 precision between the spike and spike duplicate was also acceptable with percent recoveries of 99.2 and 98.0, respectively.

RPDs between the samples and their duplicates were not able to be calculated for samples V21 and the original V23 analysis, as well as for the field blank, V31, because one or both of the determinations from each sample was less than the sample detection limit of 30.0  $\mu\text{g/L}$ . Of those samples for which precision could be determined, only V27 had an RPD greater than 20 percent.

Silver concentrations for the field blank were essentially at or less than the detection limit of 30.0  $\mu\text{g/L}$ . Preparation blanks were similarly either less than or very nearly equivalent to the detection limit of 6.00  $\mu\text{g/L}$ .

The average silver concentration for tank samples V21 and the original V23 was less than 30.0  $\mu\text{g/L}$ . The grand average for all samples was equal to or less than 47.0  $\mu\text{g/L}$  (less than values were deleted for calculations).

### SODIUM

When sample analyte concentrations exceed 1000  $\mu\text{g/ml}$  (1,000,000  $\mu\text{g/L}$ ), the normal data control limits of 75 to 125 percent recovery are not applicable to ICP analyses, as stated in the TPP, section 4.5. The sodium concentration of each sample was determined to exceed 1000  $\mu\text{g/ml}$ , consequently an alternative evaluation of accuracy was applied using serial dilution. Following serial dilution and reanalysis of each sample, a dilution RPD value was determined. The formula for this calculation follows:

$$\text{Dilution RPD} = \frac{[\text{initial conc.}] - ([\text{serial dil'n conc.}] \times \text{dilution factor})}{[\text{initial concentration}]} \times 100$$

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Using a dilution rather than a spike has advantages and disadvantages as a data quality evaluation tool. Although an evaluation of the deviation between the actually derived concentration and the expected concentration following dilution does not definitively indicate the degree of matrix interference within a sample, it does establish whether or not the analysis was performed within the linear portion of the calibration curve. Conversely, a spike is not particularly useful when the initial sample concentration is very high. To be distinguishable above the initial sample concentration, spiking must generate a final concentration at least 25 percent greater than the initial concentration, yet this frequently places the analyte concentration within the region of calibration non-linearity. The result is that percent recovery is significantly underestimated. For example, accuracy was not acceptable as indicated by spike and spike duplicate recoveries of -2884.7 and -2261.5 percent, respectively. The dilution method was applied to sodium (the only ICP analyte in which the sample concentrations exceeded 1000  $\mu\text{g/ml}$ ). Dilution RPD values less than five percent indicate that measurements are within the linear portion of the calibration curve. For all tank 107-AP samples, the dilution RPD values ranged from 0.9 to 2.6 percent, with an average of 1.52 percent. Values for each sample were as follows:

<u>Sample</u>	<u>Dilution RPD</u>
V21	1.4
V23	1.8
V26	1.3
V27	0.9
V28	1.1
V23 re-run	2.6

The recovery of all undigested LMCS standards were within control limits, ranging from 98.7 to 101.4 percent recovery. Of the non-required, digested laboratory control samples analyzed with each batch, only the LCS representing the rerun of sample V23 was not within LMCS control limits, with a 216.1 percent recovery. This indicated the presence of contamination for the LCS sample, but because such contamination was not observed elsewhere, it appeared reasonable that the accuracy of the V23 rerun was not in question.

Precision between the spike and spike duplicate was acceptable with a 1.5 relative percent difference. RPDs between the samples and their duplicates ranged from 0.3 to 9.9.

The reagent blank concentrations ranged from 263 to 699  $\mu\text{g/L}$ . Because the blanks were less than 20 percent of the sample concentrations, they were determined to not be contaminated. A remark in the chemist's narrative for sample V31, the field blank, erroneously states that the preparation blank was contaminated. V31's preparation blank had a sodium concentration which was only 11 percent of the sample and did not meet the criterion for contamination. The grand average of sample concentrations was approximately 4,300 fold greater than that of the preparation blank concentrations (527  $\mu\text{g/L}$ ).

Average sodium concentrations of the samples ranged from 2,080,000  $\mu\text{g/L}$  to 2,440,000  $\mu\text{g/L}$ , with a grand average of 2,270,000  $\mu\text{g/L}$  (2,270  $\mu\text{g/ml}$ ).

In the narrative prepared by the ICP chemist, the comment was made that contamination caused the high percent recovery (216 percent) for V23's digested LCS standard. No acceptance criteria apply to percent recovery of the digested standard, and the other sodium quality control data tend to indicate that these data are acceptable.

**ZINC**

Percent recovery of all undigested LMCS standards were within control limits, ranging from 96.8 to 104.7 percent. Three of the four digested laboratory control samples (not required) had percent recoveries which were within the LMCS control limits. The laboratory control sample representing the batch which contained samples V21 and V23 had recovery of 156.2 percent, indicating contamination in that LCS sample and potentially within the batch.

Accuracy was acceptable as indicated by the spike and spike duplicate recoveries for V21 of 76.9 percent and 78.2 percent, respectively. Accuracy for the rerun of sample V23 was also acceptable with spike and spike duplicate recoveries of 97.4 and 99.4 percent.

Precision between the spikes and spike duplicates was acceptable for both V21 and V23 with relative percent differences of 1.3 and 1.9 percent, respectively. RPDs between the samples and their duplicates were poor, ranging from 11.4 to 177.4 percent, however no acceptance criteria apply to the duplicate analyses. Sample and duplicate concentrations were approximately 68 fold greater (except for V23 rerun which was 4.5 fold greater) than the sample detection limit, consequently large percent deviations were not due to variability near the limit of instrument capability. Sample contamination is indicated as the cause of such imprecision.

An average zinc concentration of 141  $\mu\text{g/L}$  was observed in the preparation blanks representing the V21/V23 and V26-V28 batches. This was approximately 47 fold greater than the detection limit. Preparation blanks representing V21, V26 and V27 were judged to be contaminated because their concentrations were greater than 20 percent of the average sample concentration, respectively 28.5, 37.0 and 53.0 percent. The preparation blank for V31 was determined to not be contaminated, however V31, the field blank, had a average zinc concentration of 3,340  $\mu\text{g/L}$ , which was greater than any of the samples and was 223 fold greater than the sample detection limit. V31 was thus determined to be contaminated.

Average zinc concentrations in the samples ranged from 67.2  $\mu\text{g/L}$  to 2660  $\mu\text{g/L}$ , with an grand average of 1023  $\mu\text{g/L}$ . Zinc data should be considered estimates, despite the fact that no required quality control limits had failed because of the abundant evidence of contamination. The 40 fold difference between the highest and lowest average sample concentrations does not appear to be consistent with the range seen typically with the other analytes which were evaluated for tank 107-AP.

**RADIOCHEMICAL ANALYSES****GAMMA ENERGY ANALYSES (GEA)**

Samples were acid predigested and analyzed using procedure/revision number LA-548-121/D-0, and instrument numbers WB57237, WB57265, and WC38461. Samples V29 through V31 were tested on 8/24/93 and samples V32 through V34 were originally tested on 9/1/93 (holding times of 23 days and 31 days, respectively). Samples V32 through V34 were rerun on 11/18/93, a holding time of 109 days. Except for radium-226 with a maximum holding time of six months, there were no specified maximum holding times in SW-846 protocol. All GEA analytes were, therefore, within holding time criteria.

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The GEA procedure does not use an LMCS quality control standard for every isotope. The LMCS standard, which contained  $\text{Co}^{60}$  and  $\text{Cs}^{137}$  as surrogate standards, was determined with each batch. Quality control parameters for all of the GEA analytes were expressed relative to these two isotopes.

Statistically derived LMCS control limits for the  $\text{Cs}^{137}$  standard (EDP code number R901) during the period of 7/23/93 through 9/8/93 were 94.39 to 111.05 percent recovery. During that period, with 25 new observations, the average recovery was 102.84 percent, with a percent standard deviation of 1.74. Actual  $\text{Cs}^{137}$  LMCS standard recoveries for all GEA analyses were acceptable at 103.0, 102.0 and 104.0 percent as shown on the summary spread sheets.

Statistically derived LMCS control limits for the  $\text{Co}^{60}$  standard (EDP code number R905) during the period of 7/23/93 through 9/8/93 were 93.02 to 110.78 percent recovery. During that period, with 26 new observations, the average recovery was 102.82 percent, with a percent standard deviation of 1.62. Actual  $\text{Co}^{60}$  LMCS standard recoveries for all GEA analyses were acceptable at 102.0, 101.0 and 104.0 percent as shown on the summary spread sheets.

Each of the 9 GEA analytes were determined in three batches. Sample V34 (the composite of samples V21, V23, V26, V27, and V28) was analyzed twice, where each batch had a blank and LMCS standard. Sample V31 (the field blank) was analyzed in a separate batch with its blank and LMCS standard.

Because standards were not used for each analyte, it was not possible to spike the sample to determine percent recoveries of each analyte. Consequently, another quality control parameter, percent counting error was determined and was found to be acceptable for both  $\text{Co}^{60}$  and  $\text{Cs}^{137}$ . Percent counting errors ranged in value from 0.9 to 1.4 for all three batches.

Sample V34 was reanalyzed because upon evaluation of the data, the cognizant chemist determined that there was possible contamination of the blank and sample with cesium-137.

#### CERIUM-144 [includes PRASEODYMIUM-144] (by GEA)

Cerium-144 was requested in the WAP.  $\text{Pr}^{144}$  was included with  $\text{Ce}^{144}$  because in the GEA test both are measured coincidentally.  $\text{Pr}^{144}$  is the decay daughter product of  $\text{Ce}^{144}$ . The combined activity was determined from the  $\text{Pr}^{144}$  gamma energy line when the parent and daughter were at secular equilibrium.

$\text{Ce}/\text{Pr}^{144}$  was less than the sample detection limit for both the composite sample (which was less than approximately  $0.0004 \mu\text{Ci}/\text{ml}$ ), and for the field blank (which was less than approximately  $0.004 \mu\text{Ci}/\text{ml}$ ). Thus, the field blank was shown not to be contaminated.

Because  $\text{Ce}/\text{Pr}^{144}$  was less than the detection limit, it was not possible to calculate a RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**CESIUM-134 (by GEA)**

Cs<sup>134</sup> was less than the detection limit for both the composite sample (approximately less than 0.00005  $\mu\text{Ci/ml}$ ), and for the field blank (approximately less than 0.0005  $\mu\text{Ci/ml}$ ). Thus, the field blank was shown not to be contaminated.

Because Cs<sup>134</sup> was not detectable, it was not possible to calculate a RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**CESIUM-137 (by GEA)**

In the original analysis of the sample V34 (the composite) and of sample V31 (the field blank), it appeared that there may have been possible contamination of with cesium-137 based upon observation of the deviation between the sample and its duplicate and the presence of activity in a reagent blank. The rerun of V34 indicated that the sample radioactivity cesium-137 level was less than the detection limit of approximately 0.00008  $\mu\text{g/ml}$ .

Field blank Cs<sup>137</sup> was less than the detection limit of 0.000685  $\mu\text{Ci/ml}$  for the duplicate, but was 0.00104  $\mu\text{g/ml}$  for the sample. Although these data did not provide sufficient evidence to conclude whether or not the field blank was contaminated, it may be of little consequence because Cs<sup>137</sup> was not detected in the sample in three of the four determinations.

An estimated mean Cs<sup>137</sup> activity of the composite sample was calculated to be equal to or less than 0.000085  $\mu\text{Ci/ml}$  (calculated by deleting "<" values to generate discrete values).

It was not possible to calculate an RPD value as an estimate of precision between samples and their duplicates because they were less than the detection limit.

An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**COBALT-60 (by GEA)**

Co<sup>60</sup> was less than the detection limit for the composite sample and the field blank. Thus, the field blank was shown not to be contaminated. An estimated mean Co<sup>60</sup> activity of the composite sample was calculated to be equal to or less than 0.000057  $\mu\text{Ci/ml}$  (calculated by deleting "<" values to generate discrete values).

Because Co<sup>60</sup> was not detectable, it was not possible to calculate a RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**EUROPIUM-154 (by GEA)**

Eu<sup>154</sup> was less than the detection limit for the composite sample and the field blank. The field blank was shown not to be contaminated. An estimated mean Eu<sup>154</sup> activity of the composite sample was calculated to be equal to or less than 0.000155  $\mu\text{Ci/ml}$  (calculated by deleting "<" values to generate discrete values).

Because Eu<sup>154</sup> was not detectable, it was not possible to calculate an RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**EUROPIUM-155 (by GEA)**

Eu<sup>155</sup> was less than the detection limit for both the composite and field blank samples. The field blank was shown not to be contaminated. An estimated mean Eu<sup>155</sup> activity of the composite sample was calculated to be equal to or less than 0.000120  $\mu\text{Ci/ml}$  (calculated by deleting "<" values to generate discrete values).

Because Eu<sup>155</sup> was not detectable, it was not possible to calculate a sample RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**NIOBIUM-94 (by GEA)**

Nb<sup>94</sup> was less than the detection limit for the composite sample and the field blank. The field blank was shown not to be contaminated. An estimated mean Nb<sup>94</sup> activity of the composite sample was calculated to be equal to or less than 0.000046  $\mu\text{Ci/ml}$  (calculated by deleting "<" values to generate discrete values).

Because Nb<sup>94</sup> was not detectable, it was not possible to calculate a RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

**RADIUM-226 (by GEA)**

Ra<sup>226</sup> was less than the detection limit for both the composite sample and the field blank. The field blank was shown not to be contaminated. An estimated mean Ra<sup>226</sup> activity of the composite sample was calculated to be equal to or less than 0.000968  $\mu\text{Ci/ml}$  (calculated by deleting "<" values to generate discrete values).

Because Ra<sup>226</sup> was not detectable, it was not possible to calculate a RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit of Ra<sup>226</sup> is approximately 10 fold greater than most other analytes by this procedure, and consequently is not the best available procedure. Prior to generation of the TPP, the customer was aware of this limitation and stated on May 11, 1993 that the GEA procedure would be acceptable for this isotope. 222-S Laboratory could analyze for Ra<sup>226</sup> by only that procedure. The detection limit shown in the data summary table was based on the reagent blank value.

#### RUTHENIUM-106 (by GEA) [includes RHODIUM-106]

Ruthenium-106 was requested in the WAP. Rh<sup>106</sup> was included with Ru<sup>106</sup> because Ru<sup>106</sup> is detected in the presence of its daughter, Rh<sup>106</sup>. Radioactivity values were shown in the spread sheet as the sum of Rh<sup>106</sup> and Ru<sup>106</sup> activities at secular equilibrium.

Ru/Rh<sup>106</sup> was less than the detection limit for both the composite sample ( $\leq 0.000835 \mu\text{Ci/ml}$ ) and the field blank ( $< 0.000755 \mu\text{Ci/ml}$ ). The field blank was shown to not be contaminated.

Because Ru/Rh<sup>103</sup> was not detectable, it was not possible to calculate a RPD value as an estimation of precision. An evaluation of accuracy was discussed above in the general GEA discussion.

The detection limit shown in the data summary table was based on the reagent blank value.

#### TRITIUM (by LIQUID SCINTILLATION)

This procedure was performed on the direct sample using procedure/revision number LA-218-114/A-2 and instrument numbers WB27815, WC16085, and WB27818. Samples V32 through V34 were initially tested on 8/17/93 (a holding time of 16 days). Those samples were retested on 10/15/93 because the spike and spike duplicate were prepared incorrectly for the original run. A required sample holding time was not specified in SW-846 protocol for this analyte. Field blank analysis was not performed because it was not required.

There did not appear to be any analytical anomalies or difficulties during the analyses of the second run, and only the problem with the original run was with the preparation of the spikes.

Statistically derived LMCS control limits for the H<sup>3</sup> standard (EDP code number R907) during the period of 7/23/93 through 9/8/93 were 75.55 to 118.04 percent recovery. During that period, with 40 new observations, the average recovery was 97.24 percent, with a percent standard deviation of 6.81.

Percent recovery of the LMCS standard, V32, was within acceptance limits at 104.1 percent and 97.0 for the initial run and rerun, respectively. The measurement of accuracy for the first run was not possible because the spike and spike duplicate were prepared with an insufficient standard addition. For the rerun, however, both the spike and spike duplicate were within acceptance limits at 99.7 and 90.0 percent recovery, respectively.

Precision as measured by the relative percent difference between the rerun spike and spike duplicate was within acceptance limits at 5.4 percent. The RPD between the sample and its duplicate was 0.6 for both runs.

H<sup>3</sup> was detected in the initial run for the sample at 0.0335  $\mu\text{Ci/ml}$  and in the duplicate sample at 0.0337  $\mu\text{Ci/ml}$ , with a mean activity of 0.0336  $\mu\text{Ci/ml}$ . In the rerun, H<sup>3</sup> was detected in the sample at 0.0353  $\mu\text{Ci/ml}$  and in the duplicate sample at 0.0351  $\mu\text{Ci/ml}$ , with a mean activity of 0.0352  $\mu\text{Ci/ml}$ .

Sample H<sup>3</sup> activity was nearly four orders of magnitude greater than the reagent blank for the initial run, and was slightly greater than two orders of magnitude for the rerun. All blanks were less than the detection limit. Consequently, no contamination was detected in the blanks.

#### CARBON-14 (by LIQUID SCINTILLATION)

C<sup>14</sup> was performed on direct samples using procedure/revision number LA-348-104/B-0 and instrument number WB27818 on 10/14/93 for samples V32 through V34, a sample holding time of 74 days. A required maximum sample holding time is not specified in SW-846 protocol for C<sup>14</sup>. Field blank analysis was not required in the source documents and was not performed.

Statistically derived LMCS control limits for the C<sup>14</sup> standard (EDP code number R909) during the period of 7/23/93 through 12/13/93 were 61.78 to 115.72 percent recovery. During that period, with 4 new observations, the average recovery was 87.43 percent, with a percent standard deviation of 2.03.

Percent recovery of the LMCS standard, V32, was within acceptance limits at 87.0 percent. Accuracy as measured by percent recovery of the spike and spike duplicate were within acceptance limits at 87.5 and 81.1 percent recovery, respectively.

Precision as measured by the relative percent difference between the spike and spike duplicate was within acceptance limits at 7.4 percent. The RPD between the sample and its duplicate was not able to be calculated because the sample value was less than the detection limit. The blank activity was less than the detection limit, consequently, it was determined to not be contaminated.

C<sup>14</sup> was not detected in the sample (less than 0.00000218  $\mu\text{Ci/ml}$ ), but in the duplicate sample the activity was 0.00000318  $\mu\text{Ci/ml}$ . "Less than" values were calculated from the statistical detection limit.

A comment by the chemist on the batch summary sheet noted, "Sample analyte concentration is at the lower limit of detection for this method". C<sup>14</sup> activity (3,180 pCi/L in equivalent units) is approaching environmental levels.

#### SELENIUM-79 (by ION EXCHANGE/DIST/LIQUID SCINTILLATION)

Se<sup>79</sup> analysis was performed on an acid digestion instead of direct, as specified in the TPP, because acid digestion would ensure that the analyte was fully dissolved to facilitate detection. Additionally, this procedure requires an acid matrix, and the initial sample pH was basic. The analysis was done on 9/2/93 (a sample holding time of 32 days) using procedure/revision number LA-365-132/B-0 and instrument number WB27818.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

Se<sup>79</sup> activity was based upon calibration with C<sup>14</sup> because no Se<sup>79</sup> standard exists. It was only possible to generate data for a reagent blank and for the sample in duplicate on the composited sample, V34. Isotopic recovery through the preparative procedure was determined gravimetrically through the use of a carrier for both the sample and the blank. The liquid scintillation counter was calibrated for efficiency using C<sup>14</sup>, since both nuclides have approximately the same beta energy.

Carrier recoveries for the sample and duplicate were 23.0 and 28.5 percent, which were rather low. The chemist states that the low carrier recoveries were due to "initial qualification run", meaning that this was the first time that the chemist had performed this analysis and that analytical techniques were not yet refined. Another quality control parameter, the percent counting error, was acceptable, however, at 3.9 and 4.2 percent for the sample and duplicate, respectively.

The reagent blank's activity was less than the detection limit of 0.00000538  $\mu\text{Ci/ml}$ .

Analytical results for the sample and its duplicate indicate that Se<sup>79</sup> activity was less than the detection limit of 0.00000538  $\mu\text{Ci/ml}$  (or 5,380 pCi/L).

#### TECHNETIUM-99 (by EXTRACTION/LIQUID SCINTILLATION)

Tc<sup>99</sup> analyses were prepared by performing an acid predigestion of samples instead of direct analysis or acid dilution as was specified in the TPP. This alternative method was recommended because acid digestion would insure that the analyte was fully dissolved to facilitate detection. Analyses were done on 9/1/93 (a sample holding time of 31 days) using procedure/revision number LA-438-101/D-1 and instrument number WB-27818.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Statistically derived LMCS control limits for the Tc<sup>99</sup> standard (EDP code number S363) during the period of 7/23/93 through 9/8/93 were 71.90 to 135.03 percent recovery. During that period, with 36 new observations, the average recovery was 98.96 percent, with a percent standard deviation of 6.37.

Accuracy as evaluated by percent recovery of spike, spike duplicate and LMCS standard were acceptable, with values of 107.6 percent, 90.1 percent and 103.8 percent, respectively.

Precision, as measured by the relative percent difference between the spike and its duplicate was acceptable at 17.7 percent. Because the sample and duplicate Tc<sup>99</sup> activity was less than the detection limit, it was not possible to calculate an RPD value.

Tc<sup>99</sup> activity was below the detection limit of 0.0000205  $\mu\text{Ci/ml}$  for the composite sample (V34). The field blank's activity was also less than the detection limit of 0.0000363  $\mu\text{Ci/ml}$ , and was thus shown not to be contaminated.

#### STRONTIUM-90 (by SEPARATION/PROPORTIONAL COUNTING)

Sr<sup>90</sup> analyses were prepared by performing an acid predigestion of samples instead of direct analysis or acid dilution as was specified in the TPP. This alternative method was recommended because acid digestion would insure that the analyte was fully dissolved to facilitate detection. Analyses were done on 9/20/93 (a sample holding time of 50 days) using procedure/revision number LA-220-101/D-0 and instrument number WB27812.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Statistically derived LMCS control limits for the Sr<sup>90</sup> standard (EDP code number S376) during the period of 7/23/93 through 12/13/93 were 79.76 to 113.11 percent recovery. During that period, with 33 new observations, the average recovery was 98.10 percent, with a percent standard deviation of 4.73.

All quality control data were within acceptance limits. The LMCS standard had a recovery of 102.8 percent. Accuracy, as measured by the percent recovery of the spike and spike duplicate, was acceptable at 97.1 percent for both.

Precision between the spike and spike duplicate was acceptable with an RPD of 0.0 percent. The relative percent difference between the sample and duplicate was acceptable at 7.0 percent.

A field blank was not analyzed (not required), however the reagent blank was less than the detection limit of 0.0000345  $\mu\text{Ci/ml}$ .

Sr<sup>90</sup> activities for the composite sample and duplicate sample were 0.000114  $\mu\text{Ci/ml}$  and 0.000122  $\mu\text{Ci/ml}$ , respectively, with an average of 0.000118  $\mu\text{Ci/ml}$ .

#### IODINE-129 (by DISTILLATION/ION EXCHANGE/GEA)

I<sup>129</sup> analysis was performed on direct samples on 8/11/93 (a sample holding time of 10 days), using procedure/revision LA-378-103/B-2 and instrument numbers WB27818 and WC16085.

Required maximum sample holding times are not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Statistically derived LMCS control limits for the I<sup>129</sup> standard (EDP code number S928) during the period of 7/23/93 through 9/8/93 were very broad from

45.69 to 147.85 percent recovery. During that period, with four new observations, the average recovery was 93.87 percent, with a percent standard deviation of 19.03.

Accuracy as determined by the recovery of the LMCS standard was acceptable at 102.2 percent recovery. Accuracy was acceptable for both the spike and spike duplicate with percent recoveries of 94.8 and 93.3, respectively.

Precision as measured by relative percent difference between the spike and its duplicate was acceptable with 1.6 percent. Because  $I^{129}$  was less than the detection limit, it was not possible to calculate an RPD value between the sample and duplicate.

The reagent blank activity was below the detection limit of 0.0000381  $\mu\text{Ci/ml}$ .

$I^{129}$  activity was less than the detection limit for both the composite sample, less than 0.0000435  $\mu\text{Ci/ml}$ , and the sample duplicate, less than 0.0000357  $\mu\text{Ci/ml}$ .

#### NEPTUNIUM-237 (by EXTRACTION/INTERNAL PROPORTIONAL COUNTER)

$\text{Np}^{237}$  analysis was performed on direct samples on 9/2/93 (a sample holding time of 32 days), using procedure/revision LA-933-141/H-0. The instrument number was not documented on the batch sheet.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte, except that the chemist's narrative on the batch summary sheet states, "The counts on the cards for the duplicate sample (V34.8883) and the duplicate spike (V34.8083) were apparently switched. The appropriate counts were used for the calculations. Rerun unnecessary." An investigation of this situation showed that all calculations were performed correctly, but that the final values were written onto two cards which were switched. The problem was easily identified and the data were corrected by marking out the incorrect number and adding the appropriate number.

Statistically derived LMCS control limits for the  $\text{Np}^{237}$  standard (EDP code number S380) during the period of 7/23/93 through 9/8/93 were very broad from 36.87 to 112.63 percent recovery. During that period, with 7 new observations, the average recovery was 78.01 percent, with a percent standard deviation of 7.25.

Accuracy as measured by the recovery of the LMCS standard was acceptable at 81.5 percent recovery. Accuracy was acceptable for both the spike and spike duplicate with percent recoveries of 84.4 and 78.5, respectively.

Precision was acceptable based on the spike and spike duplicate with a 7.3 relative percent difference. Because  $\text{Np}^{237}$  was less than the detection limit, it was not possible to calculate a RPD value between the sample and duplicate.

The reagent blank activity was less than the detection limit of 0.0000391  $\mu\text{Ci/ml}$ , indicating that it was not contaminated.

$\text{Np}^{237}$  activity was less than the detection limit for both the composite sample and its duplicate, with values of 0.0000148  $\mu\text{Ci/ml}$  and 0.0000132  $\mu\text{Ci/ml}$ , respectively.

#### PLUTONIUM-239/240 (By ALPHA ENERGY ANALYSIS)

$\text{Pu}^{239/240}$  analysis was performed on acid digested samples on 9/2/93 (a sample holding time of 32 days), using procedure/revision LA-503-156/D-0 and instrument number WB57237.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

Statistically derived LMCS control limits for the  $\text{Pu}^{239/240}$  standard (EDP code number R211) during the period of 7/23/93 through 9/8/93 were 74.04 to 120.82 percent recovery. During that period, with 24 new observations, the average recovery was 103.09 percent, with a percent standard deviation of 4.68.

Accuracy as determined by recovery of the LMCS standard was acceptable at 106.5 percent recovery. Accuracy was acceptable for both the spike and spike duplicate with percent recoveries of 100.4 and 101.1 percent, respectively.

Precision evaluation, as a function of the relative percent difference between the spike and its duplicate, was acceptable with 0.8 RPD. Because  $\text{Pu}^{239/240}$  activity was less than the detection limit, it was not possible to calculate an RPD value between the sample and its duplicate.

The reagent blank activity was less than the detection limit of 0.000417  $\mu\text{Ci/ml}$ , indicating that it was not contaminated.

$\text{Pu}^{239/240}$  activity was less than the detection limit for both the composite sample, of less than 0.000534  $\mu\text{Ci/ml}$ , and for the sample duplicate, of less than 0.000423  $\mu\text{Ci/ml}$ . The reported sample result for  $\text{Pu}^{239/240}$  was a "less than" value using 0.05 for the  $\text{Pu}^{239/240}$  peak height.

#### PLUTONIUM-238 [not requested in WAP] (by ALPHA ENERGY ANALYSIS)

Although this analyte was not requested in the source documents, it was provided in this data package because it was generated as part of the  $\text{Pu}^{239/240}$  procedure at the subsequent request by telephone of the evaporator program.

$\text{Pu}^{238}$  analysis was performed on acid digested samples on 9/2/93 (a sample holding time of 32 days), using procedure/revision LA-503-156/D-0 and instrument number WB57237.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte.

000060

The assessment of accuracy for this method was based on the recovery of the Pu<sup>239/240</sup> LMCS standard was used because no Pu<sup>238</sup> LMCS standard was available. The recovery of this surrogate standard was acceptable with a recovery of 106.5 percent. Carrier recovery was tolerable for the sample and duplicate with percent recoveries of 75.4 and 69.2, respectively.

Because Pu<sup>238</sup> activity was less than the detection limit for the sample and its duplicate, it was not possible to calculate a RPD value between them. Assessment of precision as a function of the relative percent difference between a spike and a spike duplicate was not possible because no spikes or spike duplicate was generated.

The reagent blank activity based on Pu<sup>239/240</sup> was less than the detection limit of 0.000417  $\mu\text{Ci/ml}$ .

Composite sample and duplicate sample Pu<sup>238</sup> activity was less than the detection limits of 0.000597  $\mu\text{Ci/ml}$  and 0.000651  $\mu\text{Ci/ml}$ , respectively. The Pu<sup>238</sup> reported "less than" value was generated using 20 dpm for the Pu<sup>238</sup> sample activity.

#### AMERICIUM-241 (by ALPHA ENERGY ANALYSIS)

Am<sup>241</sup> analysis was performed on acid digested samples on 9/1/93 (a sample holding time of 31 days), and a rerun was done on 9/8/93 (a sample holding time of 38 days), using procedure/revision LA-503-156/D-0 and instrument number WB57237.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte during either batch. The rerun was performed because precision (spike/spike-duplicate RPD) was unacceptable.

Statistically derived LMCS control limits for the Am<sup>241</sup> standard (EDP code number R201) during the period of 7/23/93 through 9/8/93 were 62.61 to 128.86 percent recovery. During that period, with 31 new observations, the average recovery was 99.69 percent, with a percent standard deviation of 10.42.

Accuracy, as evaluated by the percent recovery of the LMCS standard, was acceptable with recoveries of 91.4 and 113.8 percent for the initial run and rerun, respectively. Accuracy for the initial run was acceptable for the spike with a recovery of 84.7 percent. The spike duplicate's recovery, however, was not acceptable with a percent recovery of 66.4 percent. A value of 75 percent is the minimum acceptable spike recovery. Recoveries for the rerun's spike and spike duplicate were acceptable with 82.4 and 91.8 percent, respectively.

Precision was not acceptable for the initial run's spike and spike duplicate with a relative percent difference of 24.1 percent. A precision value of 20 percent difference is the maximum acceptable relative percent difference. The RPD generated between the rerun's spike and its duplicate was acceptable with 10.7 percent. For both sets of samples and sample duplicates, the Am<sup>241</sup> activity was determined to be less than the detection limit, consequently it was not possible to calculate the relative percent differences between the pairs of samples and their duplicates.

The reagent blank activities for both runs were less than the detection limit, indicating that they were not contaminated.

From the original run, the  $\text{Am}^{241}$  activity of both the sample and sample duplicate was less than the detection limit of  $0.000637 \mu\text{Ci/ml}$ . The activity for the rerun sample and sample duplicate was less than the detection limit of  $0.0000637 \mu\text{Ci/ml}$  (a detection limit that was 10 fold lower than for the original run). These "less than" values were determined using 5 percent of the  $\text{Am}^{243}$  tracer peak as the  $\text{Am}^{241}$  peak.

#### CURIUM-244/243 (by ALPHA ENERGY ANALYSIS)

$\text{Cm}^{243/244}$  analysis was performed on acid digested samples on 9/1/93 (a sample holding time of 31 days), and a rerun was done on 9/8/93 (a sample holding time of 38 days), using procedure/revision LA-503-156/D-0 and instrument number WB57237.

A required maximum sample holding time was not specified in SW-846 protocol for this analyte, and no field blank was analyzed because it was not required in the source documents.

There did not appear to be any analytical anomalies or difficulties during the analyses of this analyte during either batch. The rerun was performed because the precision (spike/spike duplicate RPD) for  $\text{Am}^{241}$  was unacceptable.

$\text{Cm}^{243/244}$  and  $\text{Am}^{241}$  were analyzed simultaneously in the same procedure. There is, however, no standard, spike or tracer available for  $\text{Cm}^{243/244}$ . When the counter was calibrated for efficiency, it was determined that this efficiency was uniform across the entire energy counting spectrum. An approximate value for other nuclides can also be determined with the use of a tracer, such as  $\text{Am}^{243}$ .

Accuracy was measured as a function of percent recovery of the surrogate  $\text{Am}^{241}$  LMCS standard and was acceptable with recoveries of 91.4 and 113.8 percent for the original run and the rerun, respectively. Accuracy for the original run was acceptable based on the  $\text{Am}^{241}$  spike with a percent recovery of 84.7, however, the accuracy failed on the spike duplicate with a recovery of 66.4 percent. Accuracy was acceptable for the rerun with spike and spike duplicate recoveries of 82.4 and 91.8 percent, respectively. From the original run, the percent counting errors for  $\text{Cm}^{243/244}$  for the sample and sample duplicate were 32.2 and 38.2 percent, respectively.  $\text{Cm}^{243/244}$  percent counting errors from the rerun for the sample and sample duplicate were 44.9 and 41.4 percent respectively. These counting errors were based on  $\text{Am}^{241}$ .

Precision (based on  $\text{Am}^{241}$ ) was not acceptable for the initial run's spike and spike duplicate with a relative percent difference of 24.1 percent. A precision value of 20 percent difference is the maximum acceptable relative percent difference. The RPD generated between the rerun's spike and its duplicate was acceptable with 10.7 percent. For both sets of samples and sample duplicates, the  $\text{Cm}^{243/244}$  activity was determined to be less than the detection limit, consequently it was not possible to calculate the relative percent differences between the pairs of samples and their duplicates.

The reagent blank activities for both runs were less than the detection limit, indicating that they were not contaminated.

From the original run, the  $\text{Cm}^{243/244}$  activity of both the sample and sample duplicate was less than the detection limit of  $0.000637 \mu\text{Ci/ml}$ . The activity for the rerun sample and sample duplicate was less than the detection limit of  $0.0000637 \mu\text{Ci/ml}$  (a detection limit that was 10 fold lower than for the original run). These "less than" values were determined using 5 percent of the  $\text{Am}^{243}$  tracer peak as the  $\text{Cm}^{243/244}$  and  $\text{Am}^{241}$  peaks.

#### TOTAL ALPHA (by proportional counter)

Total alpha analyses were performed on acid predigested samples rather than directly or by acid dilution as was specified in the TPP. This alternative method was recommended because acid digestion would insure that the analyte was fully dissolved to facilitate detection.

The original analyses of samples V32 through V34 were done on 9/1/93 (a sample holding time of 31 days), and were rerun on 10/25/93 (a sample holding time of 85 days) using instrument numbers WB27810 and WA45709, respectively. Samples V29 through V31 were analyzed on 10/11/93 (a sample holding time of 71 days) using instrument number WB27809. All analyses were performed using procedure/revision number LA-508-101/D-0.

Detection limits and "less than" values were determined from procedure LA-508-002.

The required maximum sample holding time for total alpha activity as specified in SW-846 protocol is six months. Tank 107-AP analyses for total alpha were analyzed within the required holding time.

There did not appear to be any analytical anomalies or difficulties during these analyses, however, samples V32-V34 were reanalyzed because of unacceptable accuracy.

Statistically derived LMCS control limits for the total alpha standard (EDP code number S510) during the period of 7/23/93 through 12/13/93 were 66.12 to 132.91 percent recovery. During that period, with 75 new observations, the average recovery was 104.34 percent, with a percent standard deviation of 9.39.

LMCS standard recovery was acceptable for the original V32 through V34 batch with a recovery of 83.8 percent. The rerun of this batch had a percent recovery of 102.8, which was also acceptable. Accuracy on the LMCS standard for samples V29 through V31 was acceptable with 111.8 percent recovery. The accuracy as measured by the percent recovery of the spike and its duplicate for the original run was unacceptable, with recoveries of 68.7 and 64.3 percent, respectively. Because the accuracy was not acceptable, a rerun was performed. Accuracy for the rerun batch was mixed, where the spike recovery of 42.5 percent was unacceptable, whereas the spike duplicate was within control limits with a recovery of 87.9 percent. As directed in the QAPjP, section 3.1, paragraph 3, only one rerun will be performed when quality control parameters are repetitively exceeded. The chemist's narrative noted that possibly the spike was prepared improperly for the rerun. If the spike was indeed prepared incorrectly, then the spike duplicate's recovery (which was within the acceptable limits) may be more indicative of the quality of this run.

Precision between the V34 spike and the duplicate spike was acceptable with 6.7 RPD. For the rerun analysis, precision between the spike and its

duplicate was not acceptable, with 122.0 relative percent difference. Precision between all samples and their duplicates was unable to be calculated because all values were less than the detection limit.

Field blank activity was less than the detection limit of 0.00000196  $\mu\text{Ci/ml}$  (1960 pCi/L). The reagent blanks were uniformly less than the detection limit of approximately 0.0000014  $\mu\text{Ci/ml}$ , indicating that they were not contaminated.

Total alpha activities for the original composite sample, V34, its duplicate sample were both less than 0.00000196  $\mu\text{Ci/ml}$  (<1960 pCi/L). For the rerun, the activity levels were less than 0.000000714  $\mu\text{Ci/ml}$  (<714 pCi/L) and less than 0.00000172  $\mu\text{Ci/ml}$  (<1720 pCi/L) for the sample and its duplicate respectively. Every analytical value for V34 was less than a detection limit, however, these are estimated values because the accuracy data indicate that analytical data were biased low. Despite this limitation, total alpha activity in sample V34 is very low, and indicates the general absence of alpha radioactivity, a favorable operating condition for the 242-A evaporator.

Total alpha activity in the composite sample was approximately 100 fold greater than the maximum contaminant level for drinking water.

#### TOTAL BETA (by proportional counter)

Total beta analyses were performed on acid predigested samples on 8/24/93 (a sample holding time of 23 days) for samples V29 through V31, and on 9/1/93 (a sample holding time of 31 days) for the original run of samples V32 through V34, using procedure/revision number LA-508-101/D-0 and instrument number WB27810. On 9/8/93 (a holding time of 38 days) the rerun of samples V32 through V34 were performed by the same procedure and revision number, using instrument number WB27809.

The required maximum sample holding time for total beta activity as specified in SW-846 protocol is six months. Tank 107-AP analyses for total beta were analyzed within the required holding time.

Detection limits and "less than" values were determined from procedure LA-508-002.

There did not appear to be any analytical anomalies or difficulties during these analyses. A rerun was unnecessarily performed for samples V32 through V34 because of a data interpretation error. Due to some confusion when a new chemist replaced another who had transferred out, the new chemist mistakenly believed that the control limits for spikes were 90 to 110 percent recovery (rather than the actual limits of 75 to 125 percent). Because the spike recovery was 88.1 percent, he ordered a rerun. Both sets of data are presented in this data package.

Statistically derived LMCS control limits for the total beta standard (EDP code number S515) during the period of 7/23/93 through 9/8/93 were 81.66 to 120.68 percent recovery. During that period, with 42 new observations, the average recovery was 96.11 percent, with a percent standard deviation of 3.95.

LMCS standard recovery was acceptable for both V34 batches with percent recoveries of 95.6 and 92.5 percent. The LMCS standard recovery for the batch including sample V31 was also acceptable with a recovery of 97.4 percent.

Accuracy as measured by the percent recovery of the original total beta spike and its duplicate was acceptable with 88.1 and 93.1 percent recoveries,

respectively. For the rerun, acceptable recoveries for the spike and spike duplicate of 85.6 and 88.0 percent were generated.

Precision between the original spike and the duplicate spike was acceptable with 5.5 relative percent difference. The RPD between the rerun spike and its duplicate was 2.7 percent. Deviation between the original sample and its duplicate was rather a rather imprecise 35.0 RPD. No relative percent difference acceptance criteria were required by the WAP or QAPJP between the sample and its duplicate. The rerun's RPD between the sample and its duplicate was a reasonable 4.9 percent.

The activity of the reagent blanks were all less than the detection limit of approximately 0.00005  $\mu\text{Ci/ml}$ . The average field blank activity was 0.000387  $\mu\text{Ci/ml}$ . It would appear that the field blank was contaminated because its average activity was approximately two fold greater than that of the composite sample, however, the sample activity was less than five fold that of the detection limit. Consequently, the distinction between the activity of the field blank and the sample was minimal, where the field blank greater activity may possibly be attributed to experimental error.

The average beta activities for the original run and rerun of the composite sample were 0.0000994  $\mu\text{Ci/ml}$  and 0.000322  $\mu\text{Ci/ml}$ , respectively, with a grand average of 0.000211  $\mu\text{Ci/ml}$ .

## CONCLUSIONS

The contents of double shell Tank 107-AP appear to be relatively homogeneous. In general, inorganic analyte concentrations in each of the samples, which were collected from five separate locations within the tank, were approximately the same. It was not possible to draw such a conclusion from radiochemical analyses because the five samples were composited.

An evaluation of heterogeneity between samples was made by determining the relative percent difference between the greatest and least concentrations of the five individual samples for selected analytes (Column 2). These data are compared with the maximum relative percent difference which existed between any of the five samples and their duplicates for the corresponding analyte (Column 3). The following data shown in Table 12 are consistent with the conclusion that the constituents of tank 107-AP are reasonably homogeneous.

Table 12. Homogeneity Analysis, a Comparison of RPD Data			
Column 1 Analyte	Column 2 Max. RPD Range between highest & lowest samples	Column 3 Max. RPD between any sample and its duplicate	Column 4 Comparative Precision (Col 2 ÷ Col 3)
Specific Gravity	0.7	4.0	0.2
Aluminum	63.9	40.4	1.6
Sodium	21.2	9.9	2.1
Nitrate	7.9	2.0	4.0
Nitrite	11.1	3.1	3.6
Cyanide	25.6	22.2	1.2
Carbonate	73.5	19.3	3.8
Uranium	87.9	66.1	1.3

Analytes were selected for which the concentration was significantly greater than the detection limit and for which there were five sample locations analyzed within the tank. For the above analytes, the maximum range of relative percent differences between each of the five analyses per analyte (Column 2) was less than or equal to 4.0 fold the maximum relative percent difference between any of the five replicate analyses (Column 3). Column 4 equals Column 2 divided by Column 3. A maximum four fold difference between column 3 and column 4 is not a major difference, indicating that variability between samples was at most only four fold greater than the maximal amount of analytical precision. Because precision appears to be primarily a function of experimental error, heterogeneity in data likewise appear to be a function of experimental error and not heterogeneity of tank contents.

A summary of all data is shown in Table 13. Results shown are values derived by averaging the average results of each of the five individual samples for inorganics, or are simply the average of composite values for radiochemical analyses.

Table 13. Summary of Analytical Data for Tank 107-AP

Parameter	Results
Visual appearance	All samples were homogeneous
DSC	No exotherms were observed
Specific gravity, average	0.989
Fluoride, grand average	$\leq 101 \mu\text{g/ml}$ (estimated)
Chloride	$< 22 \mu\text{g/ml}$
Nitrate, grand average	$1,019 \mu\text{g/ml}$ (0.016M)
Nitrite, grand average	$23,500 \mu\text{g/ml}$ (0.489M)
Phosphate	$< 10 \mu\text{g/ml}$
Sulfate, grand average	$\leq 48.6 \mu\text{g/ml}$
Ammonia	$< 40 \mu\text{g/ml}$
Hydroxide	$< 250 \mu\text{g/ml}$
Cyanide, grand average	$0.58 \mu\text{g/ml}$
Carbonate, grand average	$270 \mu\text{g/ml}$
Uranium, grand average	$0.0234 \mu\text{g/ml}$
Arsenic	$< 0.0130 \mu\text{g/ml}$
Selenium	$< 0.0130 \mu\text{g/ml}$
Mercury	$< 0.003 \mu\text{g/ml}$
Aluminum, grand average	$1080 \mu\text{g/L}$
Barium, grand average	$\leq 23.9 \mu\text{g/L}$
Cadmium	$< 20 \mu\text{g/L}$
Calcium, grand average	$26,600 \mu\text{g/L}$ (estimated)
Chromium, grand average	$\leq 38.2 \mu\text{g/L}$
Iron, grand average	$\leq 219 \mu\text{g/L}$ (estimated)
Lead, grand average	$\leq 261 \mu\text{g/L}$
Magnesium, grand average	$1480 \mu\text{g/L}$ (estimated)
Manganese, grand average	$\leq 40.3 \mu\text{g/L}$
Silver, grand average	$\leq 47.0 \mu\text{g/L}$
Sodium, grand average	$2,270,000 \mu\text{g/L}$ ( $2270 \mu\text{g/ml}$ )
Zinc, grand average	$1020 \mu\text{g/L}$ (estimated)
Ce/Pr <sup>144</sup>	$< 0.0004 \mu\text{Ci/ml}$
Cs <sup>134</sup>	$< 0.00005 \mu\text{Ci/ml}$
Cs <sup>137</sup>	$\leq 0.000085 \mu\text{Ci/ml}$
Co <sup>60</sup>	$< 0.000057 \mu\text{Ci/ml}$

Table 13. Summary of Analytical Data for Tank 107-AP	
Parameter	Results
Eu <sup>154</sup>	<0.000155 $\mu\text{Ci/ml}$
Eu <sup>155</sup>	<0.000120 $\mu\text{Ci/ml}$
Nb <sup>94</sup>	$\leq$ 0.000046 $\mu\text{Ci/ml}$
Ra <sup>226</sup>	$\leq$ 0.000968 $\mu\text{Ci/ml}$
Ru/Rh <sup>106</sup>	$\leq$ 0.000835 $\mu\text{Ci/ml}$
H <sup>3</sup> , grand average	0.0344 $\mu\text{Ci/ml}$
C <sup>14</sup>	$\leq$ 0.00000268 $\mu\text{Ci/ml}$
Se <sup>79</sup>	<0.00000538 $\mu\text{Ci/ml}$
Tc <sup>99</sup>	<0.0000205 $\mu\text{Ci/ml}$
Sr <sup>90</sup>	0.000118 $\mu\text{Ci/ml}$
I <sup>129</sup>	$\leq$ 0.0000396 $\mu\text{Ci/ml}$
Np <sup>237</sup>	$\leq$ 0.0000140 $\mu\text{Ci/ml}$
Pu <sup>239/240</sup>	$\leq$ 0.000479 $\mu\text{Ci/ml}$
Pu <sup>238</sup>	$\leq$ 0.000624 $\mu\text{Ci/ml}$
Am <sup>241</sup>	<0.0000637 $\mu\text{Ci/ml}$
Cm <sup>243/244</sup>	<0.0000637 $\mu\text{Ci/ml}$
Total alpha, average	$\leq$ 0.00000122 $\mu\text{Ci/ml}$ (estimated)
Total beta, grand average	0.000211 $\mu\text{Ci/ml}$

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DATA ASSESSMENT SUMMARY TABLES

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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**SPECIFIC GRAVITY  
DIRECT ANALYSIS**

SAMPLE I.D.	LAB I.D.	RESULTS				QC RESULT			QC ID INFO.	
		SAMPLE RESULT	DUPLICATE RESULT	AVERAGE RESULT	RPD %	STD RESULT	STD % REC.	REAGENT BLANK	STD I.D.	BLANK I.D.
V21	107-AP-A	0.987	0.991	0.989	4.04	1.3876	97.9	0.979	V19	V20
V23	107-AP-B	0.989	0.992	0.990	0.30	1.3876	97.9	0.979	V19	V20
V26	107-AP-C	0.991	0.991	0.991	0.00	1.3936	98.4	0.986	V24	V25
V27	107-AP-D	0.984	0.993	0.988	0.91	1.3936	98.4	0.986	V24	V25
V28	107-AP-E	0.985	0.989	0.987	0.41	1.3936	98.4	0.986	V24	V25

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**FLUORIDE BY ION CHROMATOGRAPHY  
WATER DILUTION**

10/1/04

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.U.M. ug/ml	STD ug/ml	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	1.37E+2	1.35E+2	1.36E+2	1.47	1E+0	5.34E+1	95.4	-156.6	-152.5	3.0	<1E+0	V19	V20	V21
V23	107-AP-B	1.43E+2	1.34E+2	1.38E+2	6.5	1E+0	5.34E+1	95.4	NA	NA	NA	<1E+0	V19	V20	NA
V26	107-AP-C	1.47E+2	1.44E+2	1.46E+2	2.1	1E+0	6.01E+1	96.9	NA	NA	NA	<1E+0	V24	V25	NA
V27	107-AP-D	1.45E+2	1.44E+2	1.45E+2	0.7	1E+0	6.01E+1	96.9	NA	NA	NA	<1E+0	V24	V25	NA
V28	107-AP-E	1.42E+2	1.44E+2	1.43E+2	1.4	1E+0	6.01E+1	96.9	NA	NA	NA	<1E+0	V24	V25	NA
V31	107-AP-FB	<1.00E+0	<1.00E+0	NA	NA	1E+0	5.42E+1	96.8	NA	NA	NA	<1E+0	V29	V30	NA

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10/1/04

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.U.M. ug/ml	STD ug/ml	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	<1.10E+1	<1.10E+1	NA	NA	1E+0	6.02E+1	97.1	68.0	66.0	2.9	<1E+0	V19	V20	V21
V21	107-AP-A	<1.1E+1	<1.1E+1	NA	NA	1E+0	5.91E+1	95.3	60.8	61.8	1.6	<1E+0	V19	V20	V21

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

CHLORIDE BY ION CHROMATOGRAPHY  
WATER DILUTION

I.D.	LAB I.D.	SAMPLE ug/ml	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.LIM. ug/ml	STD ug/ml	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPKE I.D.
V21	107-AP-A	<2.2E+1	<2.2E+1	NA	NA	2E+0	7.87E+1	94.8	89.0	89.3	0.8	<2E+0	V19	V20	V21
V23	107-AP-B	<2.2E+1	<2.2E+1	NA	NA	2E+0	7.87E+1	94.8	NA	NA	NA	<2E+0	V19	V20	NA
V26	107-AP-C	<2.2E+1	<2.2E+1	NA	NA	2E+0	8.24E+1	99.3	NA	NA	NA	<2E+0	V24	V25	NA
V27	107-AP-D	<2.2E+1	<2.2E+1	NA	NA	2E+0	8.24E+1	99.3	NA	NA	NA	<2E+0	V24	V25	NA
V28	107-AP-E	<2.2E+1	<2.2E+1	NA	NA	2E+0	8.24E+1	99.3	NA	NA	NA	<2E+0	V24	V25	NA
V31	107-AP-FB	<2E+0	<2E+0	NA	NA	2E+0	7.18E+1	95.7	NA	NA	NA	<2E+0	V29	V30	NA

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**NITRATE BY ION CHROMATOGRAPHY  
WATER DILUTION**

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.U.M. ug/ml	STD ug/ml	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	9.79E+2	9.95E+2	9.87E+2	1.6	1.0E+1	5.41E+2	93.4	99.8	95.5	0.6	<1.0E+1	V19	V20	V21
V23	107-AP-B	1.01E+3	1.03E+3	1.02E+3	2.0	1.0E+1	5.41E+2	93.4	NA	NA	NA	<1.0E+1	V19	V20	NA
V26	107-AP-C	1.02E+3	1.01E+3	1.02E+3	1.0	1.0E+1	6.20E+2	97.5	NA	NA	NA	<1.0E+1	V24	V25	NA
V27	107-AP-D	1.06E+3	1.06E+3	1.06E+3	0.0	1.0E+1	6.20E+2	97.5	NA	NA	NA	<1.0E+1	V24	V25	NA
V28	107-AP-E	1.02E+3	1.00E+3	1.01E+3	2.0	1.0E+1	6.20E+2	97.5	NA	NA	NA	<1.0E+1	V24	V25	NA
V31	107-AP-FB	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.45E+2	94.1	NA	NA	NA	<1.0E+1	V29	V30	NA

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**NITRITE BY ION CHROMATOGRAPHY  
WATER DILUTION**

I.D.	LAB I.D.	SAMPLE	DUPLICATE	RESULTS		QC RESULT						QC ID INFO.			
				ug/ml	ug/ml	ug/ml	RPD %	DET.UM. ug/ml	STD ug/ml	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.
V21	107-AP-A	2.29E+4	2.29E+4	2.29E+4	0.0	1.0E+1	5.10E+2	97.0	90.4	96.1	1.1	<1.0E+1	V19	V20	V21
V23	107-AP-B	2.32E+4	2.25E+4	2.28E+4	3.1	1.0E+1	5.10E+2	97.0	NA	NA	NA	<1.0E+1	V19	V20	NA
V26	107-AP-C	2.31E+4	2.25E+4	2.28E+4	2.8	1.0E+1	5.44E+2	103.4	NA	NA	NA	<1.0E+1	V24	V25	NA
V27	107-AP-D	2.56E+4	2.51E+4	2.53E+4	2.0	1.0E+1	5.44E+2	103.4	NA	NA	NA	<1.0E+1	V24	V25	NA
V28	107-AP-E	2.39E+4	2.34E+4	2.36E+4	2.1	1.0E+1	5.44E+2	103.4	NA	NA	NA	<1.0E+1	V24	V25	NA
V31	107-AP-FB	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.40E+2	102.7	NA	NA	NA	<1.0E+1	V29	V30	NA

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

ORTHOPHOSPHATE BY ION CHROMATOGRAPHY  
WATER DILUTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.U.M. ug/ml	STD ug/ml	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.24E+2	101.4	85.4	85.1	0.2	<1.0E+1	V19	V20	V21
V23	107-AP-B	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.24E+2	101.4	NA	NA	NA	<1.0E+1	V19	V20	NA
V26	107-AP-C	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.09E+2	98.5	NA	NA	NA	<1.0E+1	V24	V25	NA
V27	107-AP-D	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.09E+2	98.5	NA	NA	NA	<1.0E+1	V24	V25	NA
V28	107-AP-E	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.09E+2	98.5	NA	NA	NA	<1.0E+1	V24	V25	NA
V31	107-AP-FB	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	5.25E+2	101.5	NA	NA	NA	<1.0E+1	V29	V30	NA

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ADDENDUM 1A

**SULFATE BY ION CHROMATOGRAPHY  
WATER DILUTION**

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		ug/ml	ug/ml	ug/ml	%	ug/ml	ug/ml	% REC	% REC	% REC	%	ug/ml			
V21	107-AP-A	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	6.06E+2	100.5	96.3	96.3	0.0	<1.0E+1	V19	V20	V21
V23	107-AP-B	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	6.06E+2	100.5	NA	NA	NA	<1.0E+1	V19	V20	NA
V26	107-AP-C	2.04E+2	2.03E+2	2.03E+2	0.5	1.0E+1	6.08E+2	100.8	NA	NA	NA	<1.0E+1	V24	V25	NA
V27	107-AP-D	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	6.08E+2	100.8	NA	NA	NA	<1.0E+1	V24	V25	NA
V28	107-AP-E	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	6.08E+2	100.8	NA	NA	NA	<1.0E+1	V24	V25	NA
V31	107-AP-FB	<1.0E+1	<1.0E+1	NA	NA	1.0E+1	6.14E+1	101.8	NA	NA	NA	<1.0E+1	V29	V30	NA

V21  
V23  
V26  
V27  
V28  
V31

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**AMMONIA  
DIRECT ANALYSIS**

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	SAMPLE DET. LIM. ug/ml	STD M	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
43 V21	107-AP-A	<8.0E+1	<4.0E+1	NA	NA	4.0E+1	4.86E-2	97.2	125.5	125.2	0.0	<8.0E+1	V19	V20	V21
43 V23	107-AP-B	<4.0E+1	<4.0E+1	NA	NA	4.0E+1	4.86E-2	97.2	NA	NA	NA	<8.0E+1	V19	V20	NA
43 V26	107-AP-C	<4.0E+1	<4.0E+1	NA	NA	4.0E+1	4.86E-2	93.5	NA	NA	NA	<4.0E+1	V24	V25	NA
43 V27	107-AP-D	<4.0E+1	<4.0E+1	NA	NA	4.0E+1	4.86E-2	93.5	NA	NA	NA	<4.0E+1	V24	V25	NA
43 V28	107-AP-E	<4.0E+1	<4.0E+1	NA	NA	4.0E+1	4.86E-2	93.5	NA	NA	NA	<4.0E+1	V24	V25	NA
43 V31	107-AP-FB	<8.0E+1	<4.0E+1	NA	NA	4.0E+1	4.86E-2	97.2	NA	NA	NA	<4.0E+1	V29	V30	NA

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

HYDROXIDE  
DIRECT ANALYSIS

I.D.	LAB I.D.	RESULTS				QC RESULT						QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPO %	SAMPLE DET. UM ug/ml	STD M	STD % REC.	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.
V21	107-AP-A	<2.5E+2	<2.5E+2	NA	NA	2.5E+2	9.22E-2	100.2	NA	NA	0.00E+0	V19	V20	NA
V23	107-AP-B	<2.5E+2	2.68E+2	NA	NA	2.5E+2	9.22E-2	100.2	NA	NA	0.00E+0	V19	V20	NA
V26	107-AP-C	<2.5E+2	<2.5E+2	NA	NA	2.5E+2	9.34E-1	101.5	NA	NA	0.00E+0	V24	V25	NA
V27	107-AP-D	<2.5E+2	<2.5E+2	NA	NA	2.5E+2	9.34E-1	101.5	NA	NA	0.00E+0	V24	V25	NA
V28	107-AP-E	<2.5E+2	<2.5E+2	NA	NA	2.5E+2	9.34E-1	101.5	NA	NA	0.00E+0	V24	V25	NA
V31	107-AP-FB	<2.5E+2	<2.5E+2	NA	NA	2.5E+2	9.42E-1	102.4	NA	NA	0.00E+0	V29	V30	NA

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

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**CYANIDE  
WATER DILUTION PREPARATION**

I.D.	LAB I.D.	SAMPLE ug/ml	RESULTS			RPD %	QC RESULT						QC ID INFO.		
			DUPLICATE ug/ml	AVERAGE ug/ml			DET.LIM. ug	STD ug/ml	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.
V21	107-AP-A	5.75E-1	5.75E-1	5.75E-1	0.0	1E-1	8.25E+2	94.5	94.7	95.0	0.3	<2.5E-2	V19	V20	V21
V23	107-AP-B	8.25E-1	8.76E-1	8.50E-1	7.7	1E-1	8.25E+2	94.5	NA	NA	NA	<2.5E-2	V19	V20	NA
V26	107-AP-C	4.84E-1	5.90E-1	5.22E-1	22.2	1E-1	8.42E+2	96.4	NA	NA	NA	<2.0E-2	V24	V25	NA
V27	107-AP-D	8.00E-1	8.20E-1	8.10E-1	3.3	1E-1	8.42E+2	96.4	NA	NA	NA	<2.0E-2	V24	V25	NA
V28	107-AP-E	5.40E-1	5.40E-1	5.40E-1	0.0	1E-1	8.42E+2	96.4	NA	NA	NA	<2.0E-2	V24	V25	NA
V31	107-AP-FB	<2.0E-2	<2.0E-2	NA	NA	1E-1	8.36E+2	95.8	NA	NA	NA	<2.0E-2	V29	V30	NA

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**TOTAL INORGANIC CARBON  
DIRECT ANALYSIS**

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I.D.	LAB I.D.	SAMPLE ug/ml	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.LIM. ug/ml	STD M	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	2.10E+2	2.55E+2	2.33E+2	19.3	5.00E+0	2.10E-1	99.1	82.1	58.9	5.2	4.80E+1	V19	V20	V21
V23	107-AP-B	4.54E+2	4.84E+2	4.59E+2	1.1	5.00E+0	2.10E-1	99.1	NA	NA	NA	4.80E+1	V19	V20	NA
V28	107-AP-C	2.38E+2	2.53E+2	2.46E+2	6.1	5.00E+0	1.92E-1	90.6	NA	NA	NA	6.00E+1	V24	V25	NA
V27	107-AP-D	2.45E+2	2.79E+2	2.62E+2	13.0	5.00E+0	1.92E-1	90.6	NA	NA	NA	6.00E+1	V24	V25	NA
V28	107-AP-E	2.51E+2	2.49E+2	2.50E+2	0.8	5.00E+0	1.92E-1	90.6	NA	NA	NA	6.00E+1	V24	V25	NA
V31	107-AP-FB	<5.00E+0	<5.00E+0	NA	NA	5.00E+0	2.01E-1	94.8	NA	NA	NA	5.80E+1	V29	V30	NA

**RE-RUNS**

82  
1A-82

I.D.	LAB I.D.	SAMPLE ug/ml	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.LIM. ug/ml	STD M	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	2.80E+2	2.80E+2	2.80E+2	0.0	5.00E+0	1.94E-1	97.0	100.6	100.8	0.2	4.20E+1	V19	V20	V21
V23	107-AP-B	2.85E+2	2.87E+2	2.86E+2	0.7	5.00E+0	1.94E-1	97.0	NA	NA	NA	4.20E+1	V19	V20	NA
V28	107-AP-C	3.01E+2	3.03E+2	3.02E+2	0.7	5.00E+0	2.09E-1	104.5	NA	NA	NA	1.30E+2	V24	V25	NA
V27	107-AP-D	2.32E+2	2.32E+2	2.32E+2	0.0	5.00E+0	2.09E-1	104.5	NA	NA	NA	1.30E+2	V24	V25	NA
V28	107-AP-E	2.49E+2	2.51E+2	2.50E+2	0.8	5.00E+0	2.09E-1	104.5	NA	NA	NA	1.30E+2	V24	V25	NA
V31	107-AP-FB	<5.00E+0	<5.00E+0	NA	NA	5.00E+0	2.15E-1	107.5	NA	NA	NA	6.20E+1	V29	V30	NA

ADD  
1-14-94

DEB.  
1/14/94

000080

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

URANIUM (TOTAL)  
DIRECT ANALYSIS

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	SAMPLE DET. UM. ug/ml	STD g/L	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	J 3.83E-2	2.93E-2	3.38E-2	28.7	6.4E-3	4.08E-2	105.7	73.3	73.3	0.0	<4.48E-3	V19	V20	V21
V23	107-AP-B	J 2.85E-2	3.41E-2	3.13E-2	18.0	5.7E-3	4.08E-2	105.7	NA	NA	NA	<4.48E-3	V19	V20	NA
V26	107-AP-C	1.83E-2	1.92E-2	1.78E-2	16.3	4.08E-3	3.88E-2	100.5	NA	NA	NA	<4.50E-3	V24	V25	NA
V27	107-AP-D	2.24E-2	2.12E-2	2.18E-2	5.5	4.48E-3	3.88E-2	100.5	NA	NA	NA	<4.50E-3	V24	V25	NA
V28	107-AP-E	3.22E-2	2.98E-2	3.10E-2	7.7	4.96E-3	3.88E-2	100.5	NA	NA	NA	<4.50E-3	V24	V25	NA
V31	107-AP-FB	2.24E-3	2.24E-3	2.24E-3	0.0	5.60E-4	3.88E-2	100.4	NA	NA	NA	5.00E-4	V29	V30	NA

*egs* 1/7/93

RERUNS

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	SAMPLE DET.UM. ug/ml	STD g/L	STD % REC	SPK % REC.	SPK DUP % REC.	SPK RPD %	REAGENT BLANK ug/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	1.79E-2	1.68E-2	1.74E-2	6.3	5.98E-3	4.54E-2	117.6	103.5	98.2	7.5	<4.50E-4	V19	V20	V21
V21	107-AP-A	2.24E-2	2.11E-2	2.18E-2	6.0	5.60E-3	3.88E-2	100.5	110.8	105.4	5.0	<4.50E-4	V19	V20	V21
V23	107-AP-B	1.50E-2	1.25E-2	1.38E-2	18.1	4.99E-3	4.54E-2	117.6	NA	NA	NA	<4.50E-4	V19	V20	NA
V23	107-AP-B	J 1.68E-2	3.34E-2	2.15E-2	66.1	5.61E-3	3.88E-2	100.5	NA	NA	NA	<4.50E-4	V19	V20	NA

11-83 83

000081

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

ARSENIC  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT								QC ID INFO.				
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.LIM. ug/ml	STD UNDIG. ug/ml	STD. UNDIG. % REC	STD ACID DIG. ug/ml	STD ACID DIG. % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD UNDIG. I.D.	STD. ACID DIG. I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.02E-1	102.2	1.39E+2	139.0	134.1	146.4	8.8	<1.3E-2	V49	V19	V20	V21
V23	107-AP-B	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.02E-1	102.2	1.39E+2	139.0	NA	NA	NA	<1.3E-2	V49	V19	V20	NA
V26	107-AP-C	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.04E-1	103.9	1.29E+2	129.1	NA	NA	NA	<1.3E-2	V51	V24	V25	NA
V27	107-AP-D	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.04E-1	103.9	1.29E+2	129.1	NA	NA	NA	<1.3E-2	V51	V24	V25	NA
V28	107-AP-E	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.04E-1	103.9	1.29E+2	129.1	NA	NA	NA	<1.3E-2	V51	V24	V25	NA
V31	107-AP-FB	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.05E-1	105.2	1.34E+2	134.2	NA	NA	NA	<1.3E-2	V53	V29	V30	NA

RE RUN

I.D.	LAB I.D.	RESULTS				QC RESULT								QC ID INFO.				
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.LIM. ug/ml	STD UNDIG. ug/ml	STD. UNDIG. % REC	STD ACID DIG. ug/ml	STD ACID DIG. % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK ug/ml	STD UNDIG. I.D.	STD. ACID DIG. I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	9.2E-2	92.4	1.25E-1	124.5	140.5	143.0	1.8	<1.3E-2	V59	V19	V20	V21
V23	107-AP-B	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	9.2E-2	92.4	1.25E-1	124.5	NA	NA	NA	<1.3E-2	V59	V19	V20	NA
V21	107-AP-A	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	9.0E-2	90.2	1.08E-1	108.0	112.3	123.1	9.2	<1.3E-2	V59	V19	V20	V21
V23	107-AP-B	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	9.0E-2	90.2	1.08E-1	108.0	NA	NA	NA	<1.3E-2	V59	V19	V20	NA

11-8484

all data U3 g/g 1/7/94

000052

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

SELENIUM  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT								QC ID INFO.				
		SAMPLE ug/ml	DUPLICATE ug/ml	AVERAGE ug/ml	RPD %	DET.LIM. ug/ml	STD UNDIG. ug/ml	STD. UNDIG. % REC.	STD ACID DIG. ug/ml	STD ACID DIG. % REC.	SPK % REC.	SPK DUP % REC.	SPK RPD %	REAGENT BLANK ug/ml	STD UNDIG. I.D.	STD. ACID DIG. I.D.	BLANK I.D.	SPIKE I.D.
V21	107-AP-A	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.03E-1	93.4	1.16E+2	116.3	107.7	111.9	3.8	<1.3E-2	V57	V19	V20	V21
V23	107-AP-B	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.03E-1	93.4	1.16E+2	116.3	NA	NA	NA	<1.3E-2	V57	V19	V20	NA
V26	107-AP-C	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.11E-1	100.6	1.23E+2	123.4	NA	NA	NA	<1.3E-2	V45	V24	V25	NA
V27	107-AP-D	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.11E-1	100.6	1.23E+2	123.4	NA	NA	NA	<1.3E-2	V45	V24	V25	NA
V28	107-AP-E	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.11E-1	100.6	1.23E+2	123.4	NA	NA	NA	<1.3E-2	V45	V24	V25	NA
V31	107-AP-FB	<1.3E-2	<1.3E-2	NA	NA	1.3E-2	1.06E-1	96.1	1.26E+2	126.4	NA	NA	NA	<1.3E-2	V47	V29	V30	NA

all data US JAF 1/7/94

85

1A-85

000083

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

MERCURY  
DIRECT ANALYSIS

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		ug/ml	ug/ml	ug/ml	%	ug/ml	ug/ml	% REC	% REC.	% REC	%	ug/ml			
V21	107-AP-A	<3E-3	<3E-3	NA	NA	3E-3	1.01E-1	100.9	99.9	95.2	4.9	<5E-3	V19	V20	V21
V23	107-AP-B	<3E-3	<3E-3	NA	NA	3E-3	1.01E-1	100.9	NA	NA	NA	<5E-3	V19	V20	NA
V26	107-AP-C	<3E-3	<3E-3	NA	NA	3E-3	1.00E-1	100.1	NA	NA	NA	<5E-3	V24	V25	NA
V27	107-AP-D	<3E-3	<3E-3	NA	NA	3E-3	1.00E-1	100.1	NA	NA	NA	<5E-3	V24	V25	NA
V28	107-AP-E	<3E-3	<3E-3	NA	NA	3E-3	1.00E-1	100.1	NA	NA	NA	<5E-3	V24	V25	NA
V31	107-AP-FB	<3E-3	<3E-3	NA	NA	3E-3	9.9E-2	98.6	NA	NA	NA	<5E-3	V29	V30	NA

dw 1/6/94

WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

86 / 86

000081

ALUMINUM  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I. D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	7.94E+2	9.35E+2	8.64E+2	16.4	2.2 E+1	4.89E+3	97.9	97.4	98.5	1.1	4.95E+1	V19	V20	V21
V23	107-AP-B	9.41E+2	1.01E+3	9.74E+2	6.9	2.2 E+1	4.89E+3	97.9	NA	NA	NA	4.95E+1	V19	V20	NA
V26	107-AP-C	1.15E+3	1.05E+3	1.10E+3	9.5	2.2 E+1	4.89E+3	97.7	NA	NA	NA	1.54E+2	V24	V25	NA
V27	107-AP-D	J 1.29E+3	8.78E+2	1.09E+3	38.3	2.2 E+1	4.89E+3	97.7	NA	NA	NA	1.54E+2	V24	V25	NA
V28	107-AP-E	J 1.26E+3	8.35E+2	1.05E+3	40.4	2.2 E+1	4.89E+3	97.7	NA	NA	NA	1.54E+2	V24	V25	NA
V31	107-AP-FB	J 8.95E+2	7.09E+2	8.02E+2	23.1	2.2 E+1	4.63E+3	92.6	NA	NA	NA	1.03E+2	V29	V30	NA
V23	107-AP-B	1.54E+3	1.29E+3	1.42E+3	17.7	2.2 E+1	4.87E+3	97.3	104.3	103.8	0.4	1.80E+2	V19	V20	V23

dw 1/6/94

87

1A-87

000085

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

BARIUM  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L/5}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	<1.50E+1	<1.50E+1	NA	NA	3.0 E+0	5.00E+3	100.1	98.9	99.5	0.6	<3.0 E+0	V19	V20	V21
V23	107-AP-B	J 1.62E+1	2.19E+1	1.90E+1	30.0	3.0 E+0	5.00E+3	100.1	NA	NA	NA	<3.0 E+0	V19	V20	NA
V26	107-AP-C	1.86E+1	2.06E+1	1.96E+1	10.6	3.0 E+0	5.00E+3	100.1	NA	NA	NA	<3.0 E+0	V24	V25	NA
V27	107-AP-D	J 1.58E+1	<1.50E+1	NA	NA	3.0 E+0	5.00E+3	100.1	NA	NA	NA	<3.0 E+0	V24	V25	NA
V28	107-AP-E	J 5.24E+1	3.68E+1	4.46E+1	35.0	3.0 E+0	5.00E+3	100.1	NA	NA	NA	<3.0 E+0	V24	V25	NA
V31	107-AP-FB	uJ <1.50E+1	3.25E+1	NA	NA	3.0 E+0	4.91E+3	98.1	NA	NA	NA	<3.0 E+0	V29	V30	NA
V23	107-AP-B	<3.00E+1	<3.00E+1	NA	NA	3.0 E+0	5.12E+3	102.5	97.5	100.1	2.7	<6.0 E+0	V19	V20	V23

dw 1/6/94

88  
JA 88

000086

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

CADMIUM  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	<2.00E+1	<2.00E+1	NA	NA	4.0 E+0	5.02E+3	100.3	98.6	98.1	0.5	<4.0 E+0	V19	V20	V21
V23	107-AP-B	<2.00E+1	<2.00E+1	NA	NA	4.0 E+0	5.02E+3	100.3	NA	NA	NA	<4.0 E+0	V19	V20	NA
V26	107-AP-C	<2.00E+1	<2.00E+1	NA	NA	4.0 E+0	4.89E+3	97.8	NA	NA	NA	<4.0 E+0	V24	V25	NA
V27	107-AP-D	<2.00E+1	<2.00E+1	NA	NA	4.0 E+0	4.89E+3	97.8	NA	NA	NA	<4.0 E+0	V24	V25	NA
V28	107-AP-E	<2.00E+1	<2.00E+1	NA	NA	4.0 E+0	4.89E+3	97.8	NA	NA	NA	<4.0 E+0	V24	V25	NA
V31	107-AP-FB	<2.00E+1	<2.00E+1	NA	NA	4.0 E+0	4.87E+3	97.4	NA	NA	NA	<4.0 E+0	V29	V30	NA
V23	107-AP-B	<4.00E+1	<4.00E+1	NA	NA	4.0 E+0	5.12E+3	102.4	93.1	96.8	3.9	<8.0 E+0	V19	V20	V23

*dw 1/6/94*

89 / 89

000087

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

CALCIUM  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I. D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	J 2.05E+4	8.99E+3	1.47E+4	78.0	8.0 E+0	5.14E+3	102.8	NA	NA	NA	3.00E+3	V19	V20	V21
V23	107-AP-B	J 4.71E+4	6.81E+4	5.76E+4	36.4	8.0 E+0	5.14E+3	102.8	NA	NA	NA	3.00E+3	V19	V20	NA
V26	107-AP-C	J 1.00E+4	1.97E+4	1.21E+4	124.3	8.0 E+0	5.27E+3	105.3	NA	NA	NA	4.31E+3	V24	V25	NA
V27	107-AP-D	J 1.04E+4	1.22E+4	1.13E+4	15.3	8.0 E+0	5.27E+3	105.3	NA	NA	NA	4.31E+3	V24	V25	NA
V28	107-AP-E	J 1.13E+5	9.97E+3	6.15E+4	167.6	8.0 E+0	5.27E+3	105.3	NA	NA	NA	4.31E+3	V24	V25	NA
V31	107-AP-FB	J 3.53E+3	1.85E+5	9.41E+4	192.5	8.0 E+0	5.07E+3	101.5	NA	NA	NA	4.02E+2	V29	V30	NA
V23	107-AP-B	J 2.59E+3	1.90E+3	2.24E+3	31.1	8.0 E+0	5.10E+3	102.0	93.6	89.5	2.8	3.10E+2	V19	V20	V23

duw 1/6/99

90

1A90

000088

ADDENDUM 1A

WHC-SD-WM-DP-053 REV 0

CHROMIUM  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	3.08E+1	<3.00 E+1	NA	NA	6.0 E+0	5.08E+3	101.5	100.3	100.3	0.0	<6.0 E+0	V19	V20	V21
V23	107-AP-B	<3.00 E+1	7.76E+1	NA	NA	6.0 E+0	5.08E+3	101.5	NA	NA	NA	<6.0 E+0	V19	V20	NA
V26	107-AP-C	<3.00 E+1	<3.00 E+1	NA	NA	6.0 E+0	4.98E+3	99.6	NA	NA	NA	<6.0 E+0	V24	V25	NA
V27	107-AP-D	<3.00 E+1	<3.00 E+1	NA	NA	6.0 E+0	4.98E+3	99.6	NA	NA	NA	<6.0 E+0	V24	V25	NA
V28	107-AP-E	<3.00 E+1	<3.00 E+1	NA	NA	6.0 E+0	4.98E+3	99.6	NA	NA	NA	<6.0 E+0	V24	V25	NA
V31	107-AP-FB	<3.00 E+1	<3.00 E+1	NA	NA	6.0 E+0	4.94E+3	98.8	NA	NA	NA	<6.0 E+0	V29	V30	NA
V23	107-AP-B	<5.50 E+1	<5.50 E+1	NA	NA	6.0 E+0	5.18E+3	103.7	97.3	100.4	3.2	<1.1 E+1	V19	V20	V23

dw 1/6/94

91

1A-91

060089

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

IRON  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	1.78E+2	1.78E+2	2.41E+2	52.4	1.10E+1	5.04E+3	100.8	98.6	97.8	0.8	4.75E+2	V19	V20	V21
V23	107-AP-B	5.81E+2	5.81E+2	5.22E+2	22.8	1.10E+1	5.04E+3	100.8	NA	NA	NA	4.75E+2	V19	V20	NA
V26	107-AP-C	<5.50E+1	<5.50E+1	NA	NA	1.10E+1	5.03E+3	100.6	NA	NA	NA	-3.57E+1	V24	V25	NA
V27	107-AP-D	<5.50E+1	<5.50E+1	NA	NA	1.10E+1	5.03E+3	100.6	NA	NA	NA	-3.57E+1	V24	V25	NA
V28	107-AP-E	3.99E+2	<5.50E+1	NA	NA	1.10E+1	5.03E+3	100.6	NA	NA	NA	-3.57E+1	V24	V25	NA
V31	107-AP-FB	2.72E+2	8.44E+2	5.58E+2	102.5	1.10E+1	4.93E+3	98.6	NA	NA	NA	3.59E+1	V29	V30	NA
V23	107-AP-B	1.78E+2	1.78E+2	2.11E+2	31.8	1.10E+1	5.06E+3	101.2	101.4	99.0	2.3	5.80E+1	V19	V20	V23

du 1/6/94

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

92

1/1/92

000050

LEAD  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	UJ <1.90E+2	3.53E+2	NA	NA	3.8 E+1	5.05E+3	101.0	108.4	103.9	4.2	<3.8 E+1	V19	V20	V21
V23	107-AP-B	5.07E+2	<1.90E+2	NA	NA	3.8 E+1	5.05E+3	101.0	NA	NA	NA	<3.8 E+1	V19	V20	NA
V26	107-AP-C	J 2.32E+2	<1.90E+2	NA	NA	3.8 E+1	4.92E+3	98.4	NA	NA	NA	<3.8 E+1	V24	V25	NA
V27	107-AP-D	<1.90E+2	<1.90E+2	NA	NA	3.8 E+1	4.92E+3	98.4	NA	NA	NA	<3.8 E+1	V24	V25	NA
V28	107-AP-E	2.00E+2	2.45E+2	2.23E+2	20.0	3.8 E+1	4.92E+3	98.4	NA	NA	NA	<3.8 E+1	V24	V25	NA
V31	107-AP-FB	<1.90E+2	<1.90E+2	NA	NA	3.8 E+1	4.91+3	98.1	NA	NA	NA	<3.8 E+1	V29	V30	NA
V23	107-AP-B	<3.20E+2	<3.20E+2	NA	NA	3.8 E+1	5.09E+3	101.8	95.9	96.4	0.5	<6.4 E+1	V19	V20	V23

*Q.P.D.*  
1-14-94

*dw 1/6/94*

93

14-93

000051

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

MAGNESIUM  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I. D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	J 8.75E+2	8.36E+2	8.56E+2	4.5	3.0 E+0	4.93E+3	98.6	NA	NA	NA	1.66E+2	V19	V20	V21
V23	107-AP-B	J 1.99E+3	1.37E+3	1.68E+3	36.3	3.0 E+0	4.93E+3	98.6	NA	NA	NA	1.66E+2	V19	V20	NA
V26	107-AP-C	J 4.68E+2	1.36E+3	9.12E+2	97.4	3.0 E+0	4.90E+3	98.0	NA	NA	NA	2.82E+2	V24	V25	NA
V27	107-AP-D	J 1.46E+3	1.66E+3	1.56E+3	13.2	3.0 E+0	4.90E+3	98.0	NA	NA	NA	2.82E+2	V24	V25	NA
V28	107-AP-E	J 6.90E+3	6.88E+2	3.80E+3	163.8	3.0 E+0	4.90E+3	98.0	NA	NA	NA	2.82E+2	V24	V25	NA
V31	107-AP-FB	J 3.75E+2	3.67E+3	2.02E+3	162.9	3.0 E+0	4.78E+3	95.5	NA	NA	NA	3.92E+1	V29	V30	NA
V23	107-AP-B	<del>1.07E+2</del> 9.79E+1	9.79E+1	1.03E+2	9.3	3.0 E+0	5.00E+3	100.0	93.8	96.3	2.5	1.14E+2	V19	V20	V23

du 1/6/94

94

11-94

000052

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

MANGANESE  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	<1.50E+1	<1.50E+1	NA	NA	3.0 E+0	5.02E+3	100.4	98.2	100.3	2.2	4.25E+0	V19	V20	V21
V23	107-AP-B	6.35E+1	7.50E+1	6.92E+1	16.6	3.0 E+0	5.02E+3	100.4	NA	NA	NA	4.25E+0	V19	V20	NA
V26	107-AP-C	$\mu\text{J}$ <1.50E+1	3.52E+1	NA	NA	3.0 E+0	4.91E+3	98.2	NA	NA	NA	8.43E+0	V24	V25	NA
V27	107-AP-D	$\mu\text{J}$ <1.50E+1	1.58E+1	NA	NA	3.0 E+0	4.91E+3	98.2	NA	NA	NA	8.43E+0	V24	V25	NA
V28	107-AP-E	$\mu\text{J}$ 1.89E+2	<1.50E+1	NA	NA	3.0 E+0	4.91E+3	98.2	NA	NA	NA	8.43E+0	V24	V25	NA
V31	107-AP-FB	$\mu\text{J}$ <1.50E+1	2.34E+2	NA	NA	3.0 E+0	4.90E+3	97.9	NA	NA	NA	<3.0 E+0	V29	V30	NA
V23	107-AP-B	<1.50E+1	<1.50E+1	NA	NA	3.0 E+0	5.06E+3	101.2	95.4	98.5	3.2	<3.0 E+0	V19	V20	V23

duw 1/6/94

95

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000053

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

SILVER  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I. D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	<3.00E+1	<3.00E+1	NA	NA	6.0 E+0	5.00E+3	99.9	103.3	102.2	1.1	<6.00E+0	V19	V20	V21
V23	107-AP-B	<3.00E+1	<3.00E+1	NA	NA	6.0 E+0	5.00E+3	99.9	NA	NA	NA	<6.00E+0	V19	V20	NA
V26	107-AP-C	J 5.09E+1	6.02E+1	5.56E+1	16.8	6.0 E+0	4.94E+3	98.9	NA	NA	NA	6.52E+0	V24	V25	NA
V27	107-AP-D	J 5.00E+1	3.68E+1	4.34E+1	30.5	6.0 E+0	4.94E+3	98.9	NA	NA	NA	6.52E+0	V24	V25	NA
V28	107-AP-E	J 5.19E+1	4.56E+1	4.87E+1	12.8	6.0 E+0	4.94E+3	98.9	NA	NA	NA	6.52E+0	V24	V25	NA
V31	107-AP-FB	UJ <3.00E+1	3.31E+1	NA	NA	6.0 E+0	4.89E+3	97.8	NA	NA	NA	<6.00E+0	V29	V30	NA
V23	107-AP-B	U 7.76E+1	6.10E+1	7.44E+1	9.1	6.0 E+0	5.09E+3	101.9	99.2	98.0	1.2	<6.00E+0	V19	V20	V23

duw 1/6/94

96

1/1-96

000054

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

SODIUM  
ACID DIGESTION

Sample I.D.		Results				QC Results								QC I.D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Dilution RPD %	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	1.98E+6	2.18E+6	2.08E+6	9.9	1.9 E+1	5.05E+3	101.1	NA	NA	NA	1.4	2.63E+2	V19	V20	V21
V23	107-AP-B	2.23E+6	2.23E+6	2.23E+6	0.3	1.9 E+1	5.05E+3	101.1	NA	NA	NA	1.8	2.63E+2	V19	V20	NA
V26	107-AP-C	2.31E+6	2.32E+6	2.32E+6	0.3	1.9 E+1	5.07E+3	101.4	NA	NA	NA	1.3	6.15E+2	V24	V25	NA
V27	107-AP-D	2.45E+6	2.43E+6	2.44E+6	0.8	1.9 E+1	5.07E+3	101.4	NA	NA	NA	0.9	6.15E+2	V24	V25	NA
V28	107-AP-E	2.34E+6	2.29E+6	2.31E+6	1.8	1.9 E+1	5.07E+3	101.4	NA	NA	NA	1.1	6.15E+2	V24	V25	NA
V31	107-AP-FB	5.16E+3	4.74E+3	4.95E+3	8.5	1.9 E+1	4.93E+3	98.7	NA	NA	NA	6.1	5.32E+2	V29	V30	NA
V23	107-AP-B	2.26E+6	2.19E+6	2.23E+6	3.1	1.9 E+1	5.04E+3	100.8	-2884.7	-2261.5	1.5	2.6	6.99E+2	V19	V20	V23

dw 1/6/94

97  
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000055

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

ZINC  
ACID DIGESTION

Sample I.D.		Results				QC Results							QC I. D.		
I.D.	Lab I.D.	Sample Concentration $\mu\text{g/L}$	Duplicate Concentration $\mu\text{g/L}$	Average Concentration $\mu\text{g/L}$	Sample Precision % RPD	Detection Limit IDL $\mu\text{g/L}$	Standard $\mu\text{g/L}$	Standard Accuracy % Rec.	Spike Accuracy % Rec.	Spike Dup Accuracy % Rec.	Spike Precision % RPD	Prepar'n Blank $\mu\text{g/L}$	Standard I.D.	Blank I.D.	Spike I.D.
V21	107-AP-A	J 5.60E+2	1.78E+2	3.69E+2	103.6	3.0 E+0	4.96E+3	99.2	76.9	78.2	1.3	1.05E+2	V19	V20	V21
V23	107-AP-B	J 2.10E+3	2.37E+3	2.23E+3	12.3	3.0 E+0	4.96E+3	99.2	NA	NA	NA	1.05E+2	V19	V20	NA
V26	107-AP-C	J 7.66E+2	8.77E+2	4.76E+2	168.3	3.0 E+0	4.94E+3	98.8	NA	NA	NA	1.76E+2	V24	V25	NA
V27	107-AP-D	J 2.34E+2	4.30E+2	3.32E+2	59.0	3.0 E+0	4.94E+3	98.8	NA	NA	NA	1.76E+2	V24	V25	NA
V28	107-AP-E	J 5.02E+3	3.01E+2	2.66E+3	177.4	3.0 E+0	4.94E+3	98.8	NA	NA	NA	1.76E+2	V24	V25	NA
V31	107-AP-FB	J 3.82E+2	6.30E+3	3.34E+3	177.1	3.0 E+0	4.84E+3	96.8	NA	NA	NA	1.44E+1	V29	V30	NA
V23	107-AP-B	7.10E+1	6.33E+1	6.72E+1	11.4	3.0 E+0	5.23E+3	104.7	97.4	99.4	1.9	1.40E+1	V19	V20	V23

dw 1/6/94

98

1A-98

000056

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

CERIUM/PRASEODYMIUM-144 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE	DUPLICATE	RESULTS		QC RESULT							QC ID INFO.		
				AVERAGE	RPD	DET.LIM.	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<4.12E-3	<4.02E-3	NA	NA	8.1E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<8.12E-4	V29	V30
V34	107-AP-COM	<4.04E-4	<3.40E-2	NA	NA	7.99E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<7.99E-5	V32	V33

RERUN

V34	107-AP-COM	<4.19 E-4	<3.79 E-4	NA	NA	8.15 E-5	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<8.15 E-5	V32	V33
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*Feb 1-7-99*

WHC-SD-MM-DP-053 REV 0  
ADDENDUM 1A

CESIUM-134 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE uCi/ml	DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<5.60E-4	<5.35E-4	NA	NA	1.3E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<1.11E-4	V29	V30
V34	107-AP-COM	<5.80E-5	<4.50E-5	NA	NA	1.05E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<1.05E-5	V32	V33

RERUN

V34	107-AP-COM	<5.25 E-5	<5.65 E-5	NA	NA	1.16 E-5	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<1.16 E-5	V32	V33
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WHCSD-WM-DP-053 REV 0

ADDENDUM 1A

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*Asst 1-2-98*

CESIUM-137 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE uCi/ml	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	1.04E-3	<6.85E-4	NA	NA	1.4E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<1.35E-4	V29	V30
V34	107-AP-COM	1.23E-4	<6.15E-5	NA	NA	1.8E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	2.61E-5	V32	V33

RERUN

V34	107-AP-COM	<8.35 E-5	<7.25 E-5	NA	NA	1.31 E-5	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<1.31 E-5	V32	V33
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*As 1-7-9*

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

COBALT-60 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE	RESULTS				QC RESULT							QC ID INFO.	
			DUPLICATE	AVERAGE	RPD	DET.LIM.	CS-137 STD	CS-137 STD	CS-137 % COUNTING	CO-60 STD	CO-60 STD	CO-60 % COUNTING	REAGENT BLANK	STD I.D.	BLANK I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% ERROR	uCi/L	% REC	% ERROR	uCi/ml		
V31	107-AP-FB	<6.35E-4	<5.55E-4	NA	NA	1.1E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<9.8E-5	V29	V30
V34	107-AP-COM	<5.55E-5	<4.19E-5	NA	NA	1.2E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<1.2E-5	V32	V33

RERUN

V34	107-AP-COM	<6.15E-5	<6.90E-5	NA	NA	1.3E-5	1.37E+2	104.0	0.9	1.06E+2	104.0	1.4	<1.3E-5	V32	V33
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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**EUROPIUM-154 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION**

I.D.	LAB I.D.	SAMPLE uCi/ml	DUPLICATE uCi/ml	RESULTS		DET.LIM. uCi/ml	QC RESULT						QC ID INFO.		
				AVERAGE uCi/ml	RPD %		CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<1.94E-3	<1.83E-3	NA	NA	3.6E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<3.0E-4	V29	V30
V34	107-AP-COM	<1.92E-4	<1.15E-4	NA	NA	3.83E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<3.83E-5	V32	V33

**RERUN**

V34	107-AP-COM	<1.88 E-4	<1.24 E-4	NA	NA	3.58 E-5	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<3.58 E-5	V32	V33
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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

**EUROPIUM-155 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION**

ID.	LAB I.D.	SAMPLE uCi/ml	DUPLICATE uCi/ml	RESULTS		QC RESULT							QC ID INFO.		
				AVERAGE uCi/ml	RPD %	DETLIM. uCi/ml	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<1.21E-3	<1.18E-3	NA	NA	2.6E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<2.49E-4	V29	V30
V34	107-AP-COM	<1.25E-4	<1.01E-4	NA	NA	2.56E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<2.56E-5	V32	V33

**RERUN**

V34	107-AP-COM	<1.29 E-4	<1.24 E-4	NA	NA	2.44 E-5	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<2.44 E-5	V32	V33
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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

NIOBIUM-94 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE uCi/ml	DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<4.96E-4	<4.96E-4	NA	NA	1.1E-4	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<1.07E-4	V29	V30
V34	107-AP-COM	<4.96E-5	<4.25E-5	NA	NA	1.12E-5	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<1.12E-5	V32	V33

RERUN

V34	107-AP-COM	<4.90 E-5	<4.44 E-5	NA	NA	9.63 E-6	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<9.63 E-6	V32	V33
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WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

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RADIUM-226 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE uCi/ml	DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<9.05E-3	<9.05E-3	NA	NA	1.6E-3	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<1.73E-3	V29	V30
V34	107-AP-COM	<9.15E-4	<1.19E-3	NA	NA	1.76E-4	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<1.76E-4	V32	V33

RERUN

V34	107-AP-COM	<8.75 E-4	<8.90 E-4	NA	NA	1.74 E-4	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<1.74 E-4	V32	V33
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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

RUTHENIUM/RHODIUM-106 BY GAMMA ENERGY ANALYSIS  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE uCi/ml	DUPLICATE uCi/ml	RESULTS		QC RESULT							QC ID INFO.		
				AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	CS-137 STD uCi/L	CS-137 STD % REC	CS-137 % COUNTING ERROR	CO-60 STD uCi/L	CO-60 STD % REC	CO-60 % COUNTING ERROR	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.
V31	107-AP-FB	<9.35E-3	<9.35E-3	NA	NA	2.0E-3	1.37E+2	103.0	0.9	1.09E+2	102.0	1.4	<1.86E-3	V29	V30
V34	107-AP-COM	<9.05E-4	<7.55E-4	NA	NA	1.63E-4	1.35E+2	102.0	0.9	1.07E+2	101.0	1.3	<1.63E-4	V32	V33

RERUN

V34	107-AP-COM	<9.50 E-4	<9.15 E-4	NA	NA	1.78 E-4	1.37 E+2	104.0	0.9	1.06 E+2	104.0	1.4	<1.78 E-4	V32	V33
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ADDENDUM 1A

WHC-SD-WM-DP-053 REV 0

TITANIUM  
DIRECT ANALYSIS

LAB	LAB I.D.	SAMPLE	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.UM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	% REC	uCi/ml			
V34	107-AP-COM	3.35E-2	3.37E-2	3.36E-2	0.8	4.45E-6	6.00E-1	104.1	3438.3	2989.3	14.0	<4.45E-6	V32	V33	V34

RERUN

LAB	LAB I.D.	SAMPLE	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.UM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	% REC	uCi/ml			
V34	107-AP-COM	3.53E-2	3.51E-2	3.52E-2	0.8	1.81E-4	7.22E-1	97.0	99.7	90.0	5.4	<1.81E-4	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

CARBON-14  
DIRECT ANALYSIS

I.D.	LAB I.D.	SAMPLE uCi/ml	RESULTS				QC RESULT						QC ID INFO.		
			DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.UM. uCi/ml	STD uCi/L	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V34	107-AP-COM	<2.18E-6	3.18E-6	NA	NA	2.17E-6	1.54E+0	87.0	87.5	81.1	7.4	<2.17E-6	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

SELENIUM -79  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS			QC RESULT				QC ID INFO.				
		SAMPLE	DUPLICATE	AVERAGE	RPO	DETUM.	STD	STD	SAMPLE	DUP	COUNTING	REAGENT	STD
V34	107-AP-COMB	uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/ml	% REC.	% REC.	% REC.	uCi/ml	I.D.	I.D.
		<5.38E-6	<5.38E-6	NA	NA	5.38E-06	NA	NA	23.0	28.5	3.9 AND 4.2	NA	V33

\* NO STANDARD OR SPIKE IS AVAILABLE FOR THIS ANALYTE

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WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

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TECHNETIUM-99  
DIRECT ANALYSIS

I.D.	LAB I.D.	SAMPLE	RESULTS				QC RESULT						QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	<2.05E-5	<2.05E-5	NA	NA	2.05E-5	4.06E+0	103.8	107.6	90.1	17.7	3.63E-5	V32	V33	V34

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WHCSD-WM-DP-053 REV 0  
ADDENDUM 1A

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STRONTIUM-90  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE uCi/ml	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	STD uCi/L	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V34	107-AP-COM	1.14E-4	1.22E-4	1.18E-4	7.0	4.05E-5	1.17E+0	102.8	87.1	87.1	0.0	3.45E-5	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

IODINE-129  
DIRECT ANALYSIS

ID.	LAB I.D.	SAMPLE uCi/ml	RESULTS			QC RESULT						QC ID INFO.			
			DUPUCATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.U.M. uCi/ml	STD uCi/ml	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V34	107-AP-COM	4.35E-5	<3.57E-5	NA	NA	3.81E-5	8.01E-1	102.2	94.8	93.3	1.6	<3.81E-5	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

NEPTUNIUM-237  
DIRECT ANALYSIS

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT						QC ID INFO.			
			DUPLICATE	AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	<1.48E-5	<1.32E-5	NA	NA	2.04E-5	1.55E+2	81.5	84.4	78.5	7.3	<3.91E-5	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

PLUTONIUM-239/240  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE uCi/ml	DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.U.M. uCi/ml	STD uCi/L	STD % REC	SPK % REC.	SPK DUP % REC	SPK RPD %	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V34	107-AP-COM	<5.34E-4	<4.23E-4	NA	NA	5.34E-4	1.39E+2	100.5	100.4	101.1	0.0	<4.17E-4	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

PLUTONIUM-238  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.	
		SAMPLE	DUPLICATE	AVERAGE	RPD	DET.UM.	STD*	STD*	SAMPLE CARRIER	DUPLICATE CARRIER	REAGENT BLANK*	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	uCi/ml			
V34	107-AP-COM	<5.97E-4	<8.51E-4	NA	NA	5.97E-4	1.39E+2	108.5	75.4	89.2	<4.17E-4	V32	V33	V34

\*BASED ON PU-239/240

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

AMERICIUM-241  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE	DUPLICATE	RESULTS		QC RESULT							QC ID INFO.		
				AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	<6.37E-4	<6.37E-4	NA	NA	6.37E-4	2.86E+1	91.4	84.7	66.4	24.1	<6.37E-4	V32	V33	V34

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I.D.	LAB I.D.	SAMPLE	DUPLICATE	RESULTS		QC RESULT							QC ID INFO.		
				AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	<6.37E-5	<6.37E-5	NA	NA	6.37E-5	3.05E+1	113.8	82.4	91.8	10.7	<6.37E-5	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

CURRUM-243/244  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT						QC ID INFO.			
			DUPLICATE	AVERAGE	RPD	DET.U.M.	STD*	STD*	SPK*	SPK DUP*	SPK RPD*	REAGENT BLANK*	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	<6.37E-4	<6.37E-4	NA	NA	6.37E-4	2.86E+1	91.4	84.7	86.4	24.1	<6.37E-4	V32	V33	V34

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RE RUN

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT						QC ID INFO.			
			DUPLICATE	AVERAGE	RPD	DET.U.M.	STD*	STD*	SPK*	SPK DUP*	SPK RPD*	REAGENT BLANK*	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	<6.37E-5	<6.37E-5	NA	NA	6.37E-5	3.05E+1	113.8	82.4	91.8	10.7	<6.37E-5	V32	V33	V34

\*Blank, Standard and Spke Values are based on Am-241.

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

TOTAL ALPHA  
ACID DIGESTION

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE uCi/ml	DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	STD uCi/L	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V31	107-AP-FB	<1.21E-6	<1.65E-6	NA	NA	2.39E-6	8.73E-3	111.8	NA	NA	NA	<9.24E-7	V29	V30	NA
V34	107-AP-COM	<1.96E-6	<1.96E-6	NA	NA	4.97E-6	7.12E-3	83.8	68.7	64.3	6.7	<1.96E-6	V32	V33	V34

RERUN

I.D.	LAB I.D.	RESULTS				QC RESULT							QC ID INFO.		
		SAMPLE uCi/ml	DUPLICATE uCi/ml	AVERAGE uCi/ml	RPD %	DET.LIM. uCi/ml	STD uCi/L	STD % REC	SPK % REC	SPK DUP % REC	SPK RPD %	REAGENT BLANK uCi/ml	STD I.D.	BLANK I.D.	SPIKE I.D.
V34	107-AP-COM	<7.14E-7	<1.72E-6	NA	NA	1.81E-6	8.03E-3	102.8	42.5	87.9	122.0	<1.30E-6	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
 ADDENDUM 1A

TOTAL BETA  
ACID DIGESTION

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V31	107-AP-FB	3.76E-4	3.98E-4	3.87E-4	5.8	2.99E-5	2.33E-1	97.4	NA	NA	NA	<2.38E-5	V29	V30	NA
V34	107-AP-COM	8.20E-5	1.17E-4	9.94E-5	35.0	8.87E-5	2.28E-1	95.8	88.1	93.1	5.5	<5.58E-5	V32	V33	V34

PBRUNS

I.D.	LAB I.D.	SAMPLE	RESULTS			QC RESULT							QC ID INFO.		
			DUPLICATE	AVERAGE	RPD	DET.LIM.	STD	STD	SPK	SPK DUP	SPK RPD	REAGENT BLANK	STD I.D.	BLANK I.D.	SPIKE I.D.
		uCi/ml	uCi/ml	uCi/ml	%	uCi/ml	uCi/L	% REC	% REC	% REC	%	uCi/ml			
V34	107-AP-COM	3.30E-4	3.15E-4	3.22E-4	4.9	7.48E-5	2.20E-1	92.5	85.8	88.0	2.7	<5.77E-5	V32	V33	V34

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WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

VALIDATION NARRATIVE

1A 122

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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**WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0**

**Tank 241-AP-107  
Data Validation Report**

Validation of the 107-AP data package was performed to the requirements provided in Sections 2.0 and 2.4 of the *Sample Management and Administration* manual (WHC-CM-5-3, Rev. 0). The data validation was performed at level "C" as defined in Section 2.0 of WHC-CM-5-3. The report forms listed in the WHC-CM-5-3 manual section 2.0 and 2.4 were not used for this report. Instead, this report has been written to provide the data user a narrative that incorporates all the required aspects that would be included on the validation forms. The overriding QA document was the *Technical Project Plan for the 222-S Laboratory in Support of the 242-A Evaporator Waste Analysis Plan (WHC-SD-WM-TPP-048 Rev.0)*. Additional guidance was given by the *242-A Evaporator Waste Analysis Plan (WAP) (WHC-SD-WM-EV-060)*. The sample analyses were performed by the Westinghouse Hanford 222-S Analytical Laboratory. Sample analyses included volatile, inorganic, and radiochemical analyses. Organic analyses were performed by Pacific Northwest Laboratories. The primary objective of the data validation effort was to ensure the usability and defensibility of the data produced for the Single Shell Tank (SST) characterization project. This was accomplished through a detailed examination of the data package to recreate the analytical process and verify that proper and acceptable analytical techniques had been applied. Additionally, the data package was checked for correct submission of required deliverables, correct data transcriptions from the raw data to the data summary forms, and for proper calculation of a number of parameters. An overall assessment of the data for each Sample Data Group (SDG) is provided on the Data Assessment Summary Form as required by WHC-CM-5-3.

Validation of the chemical analyses data package was performed to the requirements provided in Section 2.0 of WHC-CM-5-3, Rev. 0. The qualification categories for non-radiochemical analyses are presented below:

- 1 Chain of Custody
- 2 Holding Times
- 3 Instrument Calibration
- 4 Initial and Continuing Calibration Verification
- 5 Analytical Blanks
- 6 Preparation Blanks
- 7 Interference Check Sample
- 8 Laboratory Control Sample
- 9 Duplicate Analysis
- 10 Matrix Spike or Post-Digestion Spike
- 11 Retention Time
- 12 Contract Required Detection Limit Standard
- 13 Serial Dilution

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**WHC-SD-WM-DP-053**  
**ADDENDUM 1A REV. 0**

Validation of the radiochemical parameters of the data package was performed to the requirements provided in Section 2.4 of WHC-CM-5-3, Rev. 0. The unique qualification categories for radiochemical data validation are listed below:

- 1 Chain of Custody
- 2 Instrument Calibration
- 3 Efficiency Checks
- 4 Background Checks
- 5 Preparation Blanks
- 6 Laboratory Control Sample
- 7 Duplicate Analysis
- 8 Matrix Spike/Tracers/Surrogates

When Quality Assurance criteria are not met in a particular category for a sample result, the appropriate data qualifier is attached. By cross-referencing the above lists, it can be seen which qualification criteria were lacking. The RCRA validation process data qualifiers are defined as follows:

- U The material was analyzed for, but was not detected. The associated value is the MDL or SQL.
- UJ The material was analyzed for, but was not detected. The MDL or SQL is an estimated quantity.
- J The associated value is an estimated quantity.
- R The data are unusable.

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Chemical Data Validation Narrative

Inductively Coupled Plasma Spectrometry (ICP)

Total metals were determined by Inductively Coupled Plasma Spectrometry (ICP) using method LA-505-151, Rev. D-0. Sample preparation consisted of acid digestion. Major and trace elemental constituents were determined by simultaneous Inductively Coupled Argon Plasma Spectrometry (ICP) and reported in EPA CLP format using Ward Scientific Software. The ICP metal analyses were conducted in accordance with the recommended quality control requirements, with a few exceptions. Several results were qualified as estimated for exceeding control limits for duplicates, laboratory control samples, and blanks. The silver that was detected in the samples and blanks appears to have originated from carryover from relatively high level LMCS standards.

Mercury by Cold Vapor Atomic Absorption Spectrometry

Mercury was determined by cold vapor atomic absorption spectrometry (procedure LA-325-104 Rev. A-1). The hold times for mercury determination for 107-AP are critical. The maximum holding time for mercury is 13 days if stored in plastic, and 38 days if stored in glass. The mercury analyses were conducted in accordance with the recommended quality control requirements. No problems were noted for the data associated with this package.

Gaseous Hydride Atomic Absorption Spectrometry (GHAA)

Arsenic and selenium were determined by GHAA using procedure LA-355-131. The data was qualified as estimated due to problems with the LCS and ICV (>110%) recoveries. All other criteria were met.

Ammonia

Ammonia was determined by using procedure LA-634-102, Rev. D-0 and D-1. The 28 day holding time specified for ammonia was exceeded. Therefore "J" qualifiers were assigned to all samples. Ammonia was also qualified as estimated, non-detect (UJ) for spike and spike duplicate recoveries just above the control limits of 75-125%. All other QC criteria were met.

Hydroxide (OH)

Hydroxide was determined by acid-base titration using procedure LA-211-102 Rev B-0. Hydroxide (OH) was qualified as estimated (J) for exceeding the holding time. The samples were not preserved from the time of sample collection to analysis. All other QC criteria were met.

Cyanide

Cyanide was determined by distillation followed by spectrometric analysis (LA-695-102, Rev. B-0). As specified by SW-846, the holding time limit for the analysis of cyanide samples is 14 days. All samples were qualified as estimated for exceeding this limit. In addition, the duplicate analysis for sample V26 exceeded the relative percent deviation (RPD) limit (20%). As a result, sample V26 was given a "J" qualifier.

1A 126

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Ion Chromatography (IC)

Ion chromatography for fluoride was performed using procedure LA-533-105. Fluoride was qualified as estimated for missed hold times. Sample results were also qualified as estimated due to spike and spike duplicate recoveries being outside of the control limits. Subsequent re-runs yielded unacceptable recoveries. All other QC criteria were met.

Total Inorganic Carbon

Total Inorganic Carbon (TIC) was performed coulometrically using procedure LA-622-102, Rev. B-2. The original analyses were qualified due to low matrix spike and matrix spike duplicate recoveries, but the samples were re-run and acceptable data were subsequently acquired.

Total Uranium

Uranium was determined by laser fluorimetry using procedure LA-925-106. The duplicate RPD for the total uranium analysis of sample V23R1 exceeded control limits, resulting in the qualification of these results as estimated. In addition, the matrix spike recoveries for samples V21-5740 and V23-5740 were out of control limits. The samples were re-analyzed with acceptable matrix spike and MSD recoveries.

1A-127

ASD  
1-14-94

123

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Radiochemical Data Validation Narrative

Control charts for most radiochemical analyses have not been provided by the laboratory. This has resulted in qualification of the affected data as estimated. A request to provide the documentation has been made by HASM.

Americium-241 and Curium 243/244

Americium-241 was determined using procedures LA-503-156, Rev. D-0 and LA-508-051, Rev. A-3. Initially, each sample mount was counted on an Alpha Proportional Counter (APC) to determine the total activity for that mount. Americium-241 was then determined by Alpha Energy Spectrometry (AEA). A known activity of an Am-243 tracer was added to each sample mount to determine efficiency for that sample. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (<3 sigma). In this case, the background and efficiency checks were not provided. As a result, the V34 Am<sup>241</sup> sample was given a "UJ" qualifier. Curium 243/244 was determined using the same procedure and tracer and therefore was also qualified as estimated.

Plutonium 239/240

Plutonium 238, 239/240 were determined using procedure LA-503-156, Rev. D-0. Each sample mount was initially counted on an Alpha Proportional Counter (APC) to determine the total alpha activity. The mounts were then counted on the AEA detector to determine the activity of the alpha emitting isotopes. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (<3 sigma). In this case, the background and efficiency checks were not provided. As a result, the V34 Pu<sup>239/240</sup> sample was given a "UJ" qualifier. Plutonium 238 was reported since it is obtained with the Pu 239/240 data.

Neptunium 237

Neptunium 237 was determined by alpha counting using procedure LA-933-141. Each sample preparation was spiked with Np<sup>237</sup>. In this case, the background and efficiency checks were not provided. As a result, the V34 Np<sup>237</sup> sample was given a "UJ" qualifier.

Strontium-90

Strontium-90 was determined on the acid digested samples by beta counting and by using procedure LA-220-101, Rev. D-0. A SrCO<sub>3</sub> carrier is used to correct for chemical recovery; therefore, spikes are not required. Since efficiency and background check documentation was not provided with the data package, all Sr-90 results were qualified as estimated. The Sr-90 results were also qualified for preparation blank contamination above acceptable levels. All other QC criteria were met.

Iodine-129

I-129 was determined by using procedure LA-378-103. The background and efficiency checks were not provided; therefore, all results were qualified as estimated. No other problems were detected.

1A 128

124

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Technitium-99

Technitium-99 was determined by liquid scintillation using procedure LA-438-101. Quench curves and background checks were provided for all analyses. Each sample was spiked with Tc<sup>99</sup> and all quality control criteria were met.

Tritium

Tritium (H-3) was determined on the water digests by liquid scintillation (LA-218-114, Rev. A-1 and A-2). Matrix spikes and duplicates were run with each batch. Quench curves and background checks were provided with all data packages. The results were qualified as unusable for extremely high matrix spike recoveries. The tritium analysis of sample V34 was re-run because the spike was prepared incorrectly. The matrix spike recovery of the re-analysis was acceptable. All other criteria were met.

Carbon-14

Carbon-14 was also determined by liquid scintillation counting using procedure LA-348-104, Rev. B-0. Matrix spikes and duplicates were run with each batch. Quench curves and background checks were provided for each sample. All Quality Control criteria were met.

Selenium-79

Selenium 79 was determined by liquid scintillation counting using procedure LA-365-132. Matrix spikes and duplicates were run with each batch. The carrier recoveries for Selenium<sup>79</sup> were extremely low (23% and 28.5%) which resulted in the qualification of these results as unusable. In addition, an LCS was not run with the Se<sup>79</sup> analysis of sample V34, resulting in the qualification of this result as estimated. No other problems were noted.

Gamma Energy Analyses (GEA)

Gamma Energy Analysis was performed using procedure LA-548-121. No matrix spikes or tracers are required for GEA determinations. Backgrounds are run daily on each GEA detector and compared to a historical 60,000 second background. If the daily background is accepted as within limits, the 60,000 second file is used to background correct the sample data. There was possible Cs<sup>137</sup> contamination of the GEA sample; therefore, a rerun was completed and the original sample was qualified as estimated. No other problems detected with the GEA determinations.

Total Alpha and Beta

Total Alpha and Beta Analyses were performed using procedures LA-505-151, Rev. A-1 and LA-508-101, Rev. D-0. Matrix spikes are not required for Total (Gross) Beta analyses. The Total Alpha spike and spike duplicate recoveries were low and a re-run was completed. The re-run spike recovery was low and the duplicate spike recovery was not prepared properly. In result, the Total Alpha analysis was qualified as estimated, non-detect (UJ). No other problems were noted.

1A129

000125

Physical Tests

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Specific Gravity

Specific Gravity was determined using procedure LA-510-112. The 5706 samples were analyzed for SPG. The SPG RPDs were found to be well within the recognized limit (20%).

Differential Thermal

Differential Scanning Calorimetry was determined by using procedure LA-514-113, Rev. A-0. The 5711 samples were analyzed for DSC, and no problems were detected.

1A-130

000126

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A131

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

DATA ASSESSMENT FORMS

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-133

PHYSICAL DATA ASSESSMENT

DATE: 01-04-94

SAMPLE/MATRIX: V21-5711/WATER

REVIEWED BY: D.E. STROUP *per 1-7-94*

V23-5711/WATER

LABORATORY: 222-S

V26-5711/WATER

CASE #: 242A EVAPORATOR

V27-5711/WATER

SDG #: 107-AP-A-222-088

V28-5711/WATER

V21-5706/WATER

V23-5706/WATER

V26-5706/WATER

V27-5706/WATER

V28-5706/WATER

DATA ASSESSMENT

	<u>DSC</u>	<u>SPG</u>
1. <u>COC/Holding Time</u>	O	O
2. <u>LCS</u>	O	O
3. <u>Blank Analysis</u>	NA	O
4. <u>Duplicate Analysis</u>	NA	O
5. <u>Matrix Spike</u>	NA	NA

O = data had no problems

X = data qualified due to minor problems

M = data qualified due to major problems, some data may be unusable

**OVERALL ASSESSMENT:** The data is acceptable with no qualifications.

**NOTES:** The 5711 samples were analyzed for Differential Scanning Calorimetry (DSC) and and the 5706 samples were analyzed for Specific Gravity (SPG).

■Refer to the corresponding attachments for explanations of any problems.

*1A-134*

000128

**WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0**

PHYSICAL QC

Name: D.E. Stroup

Date: 01-04-94

QC Check: COC/Holding Time

COMMENTS: The samples were collected on 8-1-93. The DSC analysis was done on 8-31-93, 9-1-93, and 9-9-93 and the SPG analysis was done on 8-22-93. Holding times are 30 to 39 days for DSC and 21 days for SPG.

ACTION: No holding time criteria is specified for DSC and SPG analyses and no action is required.

*A-135*

000129

**WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0**

PHYSICAL QC

Name: D.E. Stroup

Date: 01-04-94

QC Check: LCS

COMMENTS: The LMCS recoveries for the DSC and SPG analyses are within the laboratory's control limits.

ACTION: None required.

1A-136

000130

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

PHYSICAL QC

Name: D.E. Stroup

Date: 01-04-94

QC Check: Blank Analysis

COMMENTS: No blank was available for the DSC analysis and the SPG blank results are near the expected value of pure water (1.0).

ACTION: Blank analysis is not required for DSC and no action is required.

1A-137

000131

WHC-SD-WM-DP-053  
ADDENDUM 14 REV. 0

PHYSICAL QC

Name: D.E. Stroup

Date: 01-04-94

QC Check: Duplicate Analysis

COMMENTS: No exotherms were observed for the DSC analysis and the SPG relative percent differences (RPD) range from 0 to 4.04.

ACTION: Since no exotherms were observed for the DSC analysis, duplicates were not required. The SPG RPDs are well within the recognized limit of 20 percent. No action is required.

1A-138

000132

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

PHYSICAL QC

Name: D.E. Stroup

Date: 01-04-94

QC Check: Matrix Spike

COMMENTS: Matrix spikes are not required and were not used for the DSC and SPG analyses.

ACTION: None required.

1A-139

000133

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

IC, AMMONIA, & OH<sup>-</sup> DATA ASSESSMENT

DATE: 01-05-94

SAMPLE/MATRIX: V21-7771, 5728, 5729

REVIEWED BY: D.E. STROUP *Des 1-7-94*

V23-7771, 5728, 5729

LABORATORY: 222-S

V26-7771, 5728, 5729

V27-7771, 5728, 5729

V28-7771, 5728, 5729

CASE #: 242A EVAPORATOR

V31-7771, 5728, 5729

WATER

SDG #: 107-AP-A-222-088

ASSESSMENT SUMMARY

	<u>IC</u>	<u>NH<sub>4</sub></u>	<u>OH<sup>-</sup></u>
1. <u>Chain of Custody/Holding Times</u>	X	X	X
2. <u>ICV/CCV</u>	O	O	O
3. <u>Blank Analysis</u>	O	O	O
4. <u>Matrix Spike</u>	X	X	O
5. <u>LCS</u>	O	O	O
6. <u>Duplicate Analysis</u>	O	O	O

O = data has no problems

X = data qualified due to minor problems

M = data qualified due to major problems, some data may be unusable

**OVERALL ASSESSMENT:** The data is acceptable with the minor qualifications noted above and on the corresponding attachment.

**NOTES:** The V31-xxxx samples are field blanks. Vxx-7771 samples were analyzed for IC anions, Vxx-5728 samples were analyzed for Ammonia, and Vxx-5729 samples were analyzed for Hydroxide Ions.

■ Refer to the corresponding attachments for explanations of any problems.

1A-140

000134

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

IC, AMMONIA, & OH<sup>-</sup> QC

Name: D.E. Stroup

Date: 01-05-94

QC Check: Chain of Custody/Holding Times

COMMENTS: The samples were collected on 8-1-93 and analyzed as follows:

■IC Anions: Initial analysis from 8-11-93 to 8-16-93. Re-runs on sample V21-7771 8-30-93 and 9-7-93 due to unknown peak interference with Fluoride.

The initial holding time is 10 to 15 days and the re-runs holding time is 29 to 37 days.

■Ammonia: The field blank was analyzed on 8-20-93 and the other samples were analyzed 8-30-93 and 8-31-93.

The field blank holding time is 19 days and the holding time for the remaining samples is 29 and 30 days.

■Hydroxide Ion: Analyzed on 8-11-93 for a holding time of 10 days.

SW846 does not stipulate holding criteria for IC Anions, but the EPA Method 300.0 for IC Anions analysis criteria are: Cooled to 4°C and held no longer than 28 days for Chloride, Bromide, Sulfate, and Fluoride; and no longer than 48 hours for Nitrate and Nitrite.

Ammonia holding criteria are not stated in SW846, but Standard Methods for Water and Wastewater, 17th Edition criteria are: Analyze ASAP or add H<sub>2</sub>SO<sub>4</sub> to pH <2 and refrigerate, recommended holding time of 7 days and EPA regulatory maximum holding time of 28 days.

Hydroxide Ion analysis holding criteria are not given in SW846, Standard Methods, or EPA Methods, but the samples probably need to be handled the same as alkalinity specimens which require refrigeration and analysis as soon as possible.

No preservation techniques were applied to the samples from the time of collection to analysis. The IC Anions analysis for Nitrate and Nitrite exceeded the required 48 hours, the IC Anions Fluoride re-runs exceeded the required 28 days, and the Ammonia analysis, except the field blank, exceeded the required 28 days.

ACTION: Qualify the IC Anions positive results as estimated (J) and non detects as estimated, non detect (UJ). Qualify the Ammonia and Hydroxide results as estimated, non detect (UJ).

<u>Sample</u>	<u>Constituent</u>	<u>Value/Qualifier</u>
All	All IC Anions	See Attached Data Sheets
All	Ammonia	See Attached Data Sheet
All	Hydroxide	See Attached Data Sheet

1A-101

000135

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

IC, AMMONIA, & OH<sup>-</sup> QC

Name: D.E. Stroup

Date: 01-05-94

QC Check: ICV/CCV

COMMENTS: There is no initial or continuing calibration information included in the data package. The retention times for the IC Anions analysis remained within 10 percent of the LMCS run.

ACTION: The accuracy and precision for the IC Anions and Hydroxide analyses are within the laboratory's control limits. The Ammonia spike and spike duplicate recoveries are just above the control limits of 75 to 125 percent (125.5 and 125.2 percent). The instrumentation used for the analyses functioned within the laboratory's control limits and no action is required.

1A-142

000136

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

IC, AMMONIA, & OH<sup>-</sup> QC

Name: D.E. Stroup

Date: 01-05-94

QC Check: Blank Analysis

COMMENTS: The field blank and reagent blank results are non detect for the analyses included in this assessment.

ACTION: No action is required.

1A 143

000137

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

IC, AMMONIA, & OH<sup>-</sup> QC

Name: D.E. Stroup

Date: 01-05-94

QC Check: Matrix Spike

COMMENTS: The Fluoride spike and spike duplicate recoveries are not within the control limits due to interference from an unknown coeluting peak. Subsequent re-runs yielded unacceptable recoveries. The Ammonia spike and spike duplicate recoveries are just above control limits of 75 to 125 percent (125.5 & 125.2). The Hydroxide recoveries are within the control limits.

ACTION: Qualify the Fluoride results as estimated (J) and the V21 re-run results as estimated, non detect (UJ). Qualify the Ammonia results as estimated, non detect (UJ).

<u>Sample</u>	<u>Constituent</u>	<u>Value/Qualifier</u>
All	Fluoride	See Attached Data Sheets
V21 Rerun	All	See Attached Data Sheet
All	Ammonia	See Attached Data Sheet

1A 100

000138

**WHC-SD-WM-DP-053**  
**ADDENDUM 1<sup>A</sup> REV. 0**

IC, AMMONIA, & OH<sup>-</sup> QC

Name: D.E. Stroup

Date: 01-05-94

QC Check: LCS

COMMENTS: The LMCS recoveries for the analyses included in this assessment are within the laboratory's control limits.

ACTION: No action is required.

1A-145

000139

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

IC, AMMONIA, & OH<sup>-</sup> QC

Name: D.E. Stroup

Date: 01-05-94

QC Check: Duplicate Analysis

COMMENTS: All of the relative percent differences (RPD) are within 20 percent or are not calculable due to results below the detection limits.

ACTION: No action is required.

1A-146

000140

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

CYANIDE DATA ASSESSMENT

DATE	<u>01-05-94</u>	SAMPLES/MATRIX	<u>V-21/WATER</u>
REVIEWED BY	<u>D.E. Berkowitz</u> <i>DEB 1/5/94</i>		<u>V-23/WATER</u>
LABORATORY	<u>WHC-222S</u>		<u>V-26/WATER</u>
CASE #	<u>242-Evaporator</u>		<u>V-27/WATER</u>
SDG #	<u>241-AP-107</u>		<u>V-28/WATER</u>
			<u>V-31/WATER</u>
			<u>                  </u>
			<u>                  </u>

DATA ASSESSMENT SUMMARY

	<u>CN</u>
1. <u>Chain of Custody/Holding Times</u>	<u>X</u>
2. <u>Instrument Calibration</u>	<u>0</u>
3. <u>ICV/CCV Standards</u>	<u>0</u>
4. <u>Blanks</u>	<u>0</u>
5. <u>Laboratory Control Sample</u>	<u>0</u>
6. <u>Duplicate Analysis</u>	<u>X</u>
7. <u>Matrix Spike/Matrix Spike Dup.</u>	<u>0</u>
8. <u>Other Quality Control</u>	<u>NA</u>

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The data is acceptable with the minor qualifications noted above and on the corresponding quality control attachments.

NOTES: None

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*1A-107*

000141

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: HOLDING TIMES

COMMENTS: Analytical holding times were assessed to determine whether the requirements for CN analyses were met. The maximum holding times are 14 days. The samples were collected on 08/01/93. Analyses for all samples except V-31 were conducted on 08/30/93. Sample V-31 was analyzed on 08/31/93. Holding times are 29 and 30 days respectively. All samples were received in good condition.

ACTION: The holding time for cyanide analysis is 14 days.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-21	CN	0.575 ug/ml J
V-23	CN	0.650 J
V-26	CN	0.522 J
V-27	CN	0.610 J
V-28	CN	0.540 J
V-31	CN	<0.020 UJ

1A 148

000142

DER 1/5/94

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: INSTRUMENT CALIBRATION

COMMENTS: The data was examined to determine whether the instruments used were calibrated at the correct frequency and that the calibration was performed correctly. All instruments must be calibrated on a daily basis or upon each set-up. Data is qualified as unusable if reported from an instrument that was not calibrated.

ACTION: All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-149

000143

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: INITIAL AND CONTINUING CALIBRATION VERIFICATION

**COMMENTS:** An Initial Calibration Verification (ICV) standard must be run at the beginning of each run. A Continuing Calibration Verification (CCV) standard must be run at a 10% frequency. The recoveries for all ICVs and CCVs must be within  $\pm 10\%$  of the true value. If the ICV/CCV results are outside the acceptable range, all associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-150

000144

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: BLANKS

**COMMENTS:** Calibration and preparation blanks were evaluated for the presence of contaminants. Calibration blanks should be run at a 10% frequency. All analytes exhibiting a concentration  $\leq 5$  times the corresponding blank result shall be qualified as non-detects. If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-151

000145

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: LABORATORY CONTROL STANDARD

**COMMENTS:** The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. All LCS results must fall within the control limits of  $\pm 20\%$  of the true value. If the LCS recovery is  $> 120\%$  or  $50 - 79\%$ , sample results are qualified as estimated. Results associated with an LCS recovery of  $< 50\%$  are qualified as unusable.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-152

000146

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are indicators of laboratory precision based on each sample matrix. Duplicate analysis must be performed at a 5% frequency or 1 per batch, whichever is greater. The relative percent deviation (RPD) for duplicate analyses should be less than 20% for sample results greater than 10 times the IDL. If the RPD is greater than 20%, the associated sample results are qualified as estimated.

**ACTION:** With the exception of sample V-26 all criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-26	CN	0.522 ug/ml J

1A 153

000147

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

CYANIDE QC

Name D.E. Berkowitz

Date 01-05-94

QC Check: MATRIX SPIKE/MATRIX SPIKE DUPLICATE

COMMENTS: Matrix spike sample analyses provide information about the effect of each sample matrix on the digestion and measurement methodology. Matrix spikes must be performed at a 20% frequency and recoveries should be between 75-125%. If the spike result is between 30-74% or >125%, results are qualified as estimated. Sample results associated with a spike recovery of less than 30% are qualified as unusable.

ACTION: All criteria were met.

sample #      constituent      value/qualifier

1A-154

000148

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A 155

000149

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-156

000150

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-157

000151

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-158

000152

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

TOTAL INORGANIC CARBON DATA ASSESSMENT

DATE	<u>01-04-94</u>	SAMPLES/MATRIX	<u>V-21/WATER</u>
REVIEWED BY	<u>D.E. Berkowitz</u> <i>DEB for</i>		<u>V-23/WATER</u>
LABORATORY	<u>WHC-222S</u> <i>D.E. Berkowitz</i>		<u>V-26/WATER</u>
CASE #	<u>242-Evaporator</u> <i>1-14-94</i>		<u>V-27/WATER</u>
SDG #	<u>241-AP-107</u>		<u>V-28/WATER</u>
			<u>V-31/WATER</u>
			<u>                  </u>
			<u>                  </u>

DATA ASSESSMENT SUMMARY

	<u>TIC</u>	
1. <u>Chain of Custody/Holding Times</u>	<u>0</u>	
2. <u>Instrument Calibration</u>	<u>0</u>	
3. <u>ICV/CCV Standards</u>	<u>0</u>	
4. <u>Blanks</u>	<u>0</u>	
5. <u>Laboratory Control Sample</u>	<u>0</u>	
6. <u>Duplicate Analysis</u>	<u>0</u>	
7. <u>Matrix Spike/Matrix Spike Dup.</u>	<u><del>0</del>X</u>	<i>DEB 1-14-94</i>
8. <u>Other Quality Control</u>	<u>NA</u>	

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The data is acceptable.

NOTES: None

1A 159

000153

1-14-94

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

TOTAL INORGANIC CARBON QC

Name D.E. Berkowitz

Date 01-04-94

QC Check: HOLDING TIMES

**COMMENTS:** Analytical holding times were assessed to determine whether the requirements for Total Inorganic Carbon analyses were met. The maximum holding time for the analysis is 28 days. On 08-01-93 the samples were collected and the last analysis was conducted on 08-20-93. All samples were received in good condition and preserved in accordance with SW-846.

**ACTION:** No action was required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 160

060154

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
TOTAL INORGANIC CARBON QC

Name D.E. Berkowitz

Date 01-04-94

QC Check: INSTRUMENT CALIBRATION

**COMMENTS:** The data was examined to determine whether the instruments used were calibrated at the correct frequency and that the calibration was performed correctly. All instruments must be calibrated on a daily basis or upon each set-up. The data is qualified as unusable if reported from an instrument that was not calibrated or was calibrated with less than the minimum number of standards.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-161

000155

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

TOTAL INORGANIC CARBON QC

Name D.E. Berkowitz

Date 01-04-94

QC Check: INITIAL AND CONTINUING CALIBRATION VERIFICATION

**COMMENTS:** An Initial Calibration Verification (ICV) standard must be run at the beginning of each run. A Continuing Calibration Verification (CCV) standard must be run at a 10% frequency. The recoveries for all ICVs and CCVs must be within  $\pm 10\%$  of the true value. If the ICV/CCV results are outside the acceptable range, all associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 162

000156

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

TOTAL INORGANIC CARBON QC

Name D.E. Berkowitz

Date 01-04-94

QC Check: BLANKS

**COMMENTS:** Calibration and preparation blanks were evaluated for the presence of contaminants. Calibration blanks should be run at a 10% frequency. Analytes exhibiting a concentration of less than 5 times the corresponding blank shall be qualified as non-detects (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** No action required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 163

000157

DEC. 11/14/94

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

TOTAL INORGANIC CARBON QC

Name D.E. Berkowitz

Date 01-04-94

QC Check: LABORATORY CONTROL STANDARD

**COMMENTS:** The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. All LCS results must fall within the control limits of  $\pm 20\%$  of the true value. If the LCS recovery is  $> 120\%$  or  $50 - 79\%$ , sample results are qualified as estimated. Results associated with an LCS recovery of  $< 50\%$  are qualified as unusable.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 164

000158

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

TOTAL INORGANIC CARBON QC

Name D.E. Berkowitz

Date 01-04-94

QC Check: DUPLICATE ANALYSIS

COMMENTS: Duplicate analyses are indicators of laboratory precision based on each sample matrix. Duplicate analysis must be performed at a 5% frequency or 1 per batch, whichever is greater. The relative percent deviation (RPD) for duplicate analyses should be less than 20% for sample results greater than 10 times the IDL. If the RPD is greater than 20%, the associated sample results are qualified as estimated.

ACTION: All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-165

000159

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
TOTAL INORGANIC CARBON QC

Name D. E. Berkowitz

Date 01-04-94

QC Check: MATRIX SPIKE/MATRIX SPIKE DUPLICATE

**COMMENTS:** Matrix spike analyses are evaluated in order to verify the accuracy of the reported data, and recoveries should be between 75 and 125%. If a spike result is between 30 and 74%, the corresponding sample data are qualified as estimated. Sample results associated with a spike recovery of less than 30% are qualified as unusable.

**ACTION:** The results affiliated with the initial set of analyses were considered to be estimated due to low matrix spike and matrix spike duplicate recoveries. As a consequence, the samples were re-run, and acceptable data were subsequently acquired. The original results were qualified as indicated in the following table.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V21	TIC	210 ug/ml J
V23	TIC	454 J
V26	TIC	238 J
V27	TIC	245 J
V28	TIC	251 J
V31	TIC	< 5.00 UJ

1A-166

AD  
1-14-94  
160

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-167

000161

WHC-SD-WM-DP-053  
ADDENDUM 1/REV. 0

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*1A-168*

000162

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-169

000163

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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*1A-170*

000164

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

WET CHEMISTRY DATA ASSESSMENT

DATE	<u>01-07-94</u>	SAMPLES/MATRIX	<u>V21-5740/WATER</u>
REVIEWED BY	<u>M.I. Weyns-Rolloson</u> <i>dw</i>	<u>1/7/94</u>	<u>V21R1-5740/WATER</u>
LABORATORY	<u>222-S</u>		<u>V21R2-5740/WATER</u>
CASE #	<u>242 EVAPORATOR</u>		<u>V23-5740/WATER</u>
SDG #	<u>241-AP-107</u>		<u>V23R1-5740/WATER</u>
			<u>V23R2-5740/WATER</u>
			<u>V26-5740/WATER</u>
			<u>V27-5740/WATER</u>
			<u>V28-5740/WATER</u>
			<u>V31-5740/WATER</u>

DATA ASSESSMENT SUMMARY

	<u>TOTAL</u>
	<u>U</u>
1. <u>Chain of Custody/Holding Times</u>	<u>0</u>
2. <u>Instrument Calibration</u>	<u>0</u>
3. <u>ICV/CCV Standards</u>	<u>0</u>
4. <u>Blanks</u>	<u>0</u>
5. <u>Laboratory Control Sample</u>	<u>0</u>
6. <u>Duplicate Analysis</u>	<u>X</u>
7. <u>Matrix Spike/Matrix Spike Dup.</u>	<u>X</u>
8. <u>Other Quality Control</u>	<u>N/A</u>

0 = data had no problems

X = minor problems, data may be qualified

M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The total uranium chemical analyses were conducted in accordance with the recommended quality control requirements with a few minor qualifications for exceeding control limits with duplicate and matrix spike analyses.

NOTES: The total uranium analyses were performed by laser fluorimetry using procedure LA-925-106.

*1A-171*

000165

WHC-SD-WM-DP-053  
ADDENDUM 1AREV. 0

Wetchem QC

Name M.I. Weyns-Rolloson

Date 01-07-94

QC Check: HOLDING TIMES

**COMMENTS:** Analytical holding times were assessed to determine whether the requirements for wet chemistry analyses were met. The samples were collected on 08/01/93 and received by TMA Laboratory on 08/02/93. Analyses were conducted between 9-9-93 and 11-1-93. Since no holding times have been established for total uranium, the holding times are accepted. All samples were received in good condition and preserved in accordance with SW-846.

**ACTION:** No action was required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-172

000166

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Wetchem QC

Name M.I. Weyns-Rollosson

Date 01-07-94

QC Check: INSTRUMENT CALIBRATION

**COMMENTS:** The data was examined to determine whether the instruments used were calibrated at the correct frequency and that the calibration was performed correctly. All instruments must be calibrated on a daily basis or upon each set-up. Ion Chromatography calibration must be performed with a minimum of a blank and 3 standards with a minimum correlation coefficient of 0.995. Data is qualified as unusable if reported from an instrument that was not calibrated or was calibrated with less than the minimum number of standards. Associated sample results were qualified as estimated if the correlation coefficient was less than 0.995.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-173

000167

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
Wetchem OC

Name M.I. Weyns-Rolloson

Date 01-07-94

QC Check: INITIAL AND CONTINUING CALIBRATION VERIFICATION

**COMMENTS:** An Initial Calibration Verification (ICV) standard must be run at the beginning of each run. A Continuing Calibration Verification (CCV) standard must be run at a 10% frequency. The recoveries for all ICVs and CCVs must be within  $\pm 10\%$  of the true value. If the ICV/CCV results are outside the acceptable range, all associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-174

000168

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Wetchem QC

Name M.I.Weyns-Rolloson

Date 01-07-94

QC Check: BLANKS

**COMMENTS:** Calibration and preparation blanks were evaluated for the presence of contaminants. Calibration blanks should be run at a 10% frequency. If the concentration of analytes in the blanks exceeded 5 times the sample concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-175

000169

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Wetchem QC

Name M.I.Weyns-Rollosson

Date 01-07-94

QC Check: LABORATORY CONTROL SAMPLE

**COMMENTS:** The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. All LCS results must fall within the control limits of  $\pm 20\%$  of the true value. If the LCS recovery is  $> 120\%$  or  $50 - 79\%$ , sample results are qualified as estimated. Results associated with an LCS recovery of  $< 50\%$  are qualified as unusable.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-176

000170

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Wetchem QC

Name M.I.Weyns-Rolloson

Date 01-07-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are indicators of laboratory precision based on each sample matrix. Duplicate analysis must be performed at a 5% frequency or 1 per batch, whichever is greater. The relative percent deviation (RPD) for duplicate analyses should be less than 20% for sample results greater than 10 times the IDL. If the RPD is greater than 20%, the associated sample results are qualified as estimated.

**ACTION:** The duplicate RPD for the total uranium analysis of sample V23R1 exceeded control limits, resulting in the qualification of this results as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value(ug/ml)/qualifier</u>	
V23R2-5740	U	1.68E-2	J

1A-177

000171

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

Wetchem QC

Name M.I. Weyns-Rolloson

Date 01-07-94

QC Check: MATRIX SPIKE/MATRIX SPIKE DUPLICATE

**COMMENTS:** Matrix spike sample analyses provide information about the effect of each sample matrix on the digestion and measurement methodology. Matrix spikes must be performed at a 20% frequency and recoveries should be between 75-125%. If the spike result is between 30-74% or >125%, results are qualified as estimated. Sample results associated with a spike recovery of less than 30% are qualified as unusable.

**ACTION:** The matrix spike recoveries for samples V21-5740 and V23-5740 were out of control limits, resulting in the qualification of these results as estimated. The samples were re-analyzed with acceptable matrix spike and MSD recoveries.

<u>sample #</u>	<u>constituent</u>	<u>value(ug/ml)/qualifier</u>	
V21-5740	U	3.83E-2	J
V23-5740	U	2.85E-2	J

1A-178

000172

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

ICP DATA ASSESSMENT

DATE	<u>1-5-94</u>	SAMPLES/MATRIX	<u>V21/WATER</u>
REVIEWED BY	<u>J.M. JONES</u> <i>JMJ 1-29-94</i>		<u>V23/WATER</u>
LABORATORY	<u>222-S</u>		<u>V26/WATER</u>
CASE #	<u>242 EVAPORATOR</u>		<u>V27/WATER</u>
SDG #	<u>241-AP-107</u>		<u>V28/WATER</u>
			<u>V31/WATER</u>

DATA ASSESSMENT SUMMARY

	<u>AA</u>			
1. <u>Chain of Custody/Holding Times</u>	<u>0</u>			
2. <u>Instrument Calibration</u>	<u>0</u>			
3. <u>ICV/CCV Standards</u>	<u><del>M</del> X</u>	<i>ADD 1-14-94</i>		
4. <u>Blanks</u>	<u>0</u>			
5. <u>Interference Check Sample</u>	<u>N/A</u>			
6. <u>Laboratory Control Sample</u>	<u>M</u>			
7. <u>Duplicate Analysis</u>	<u>0</u>			
8. <u>Matrix Spike/Matrix Spike Dup.</u>	<u>0</u>			
9. <u>CRDL Standard</u>	<u>N/A</u>			
10. <u>Serial Dilution</u>	<u>N/A</u>			
11. <u>Other Quality Control</u>	<u>N/A</u>			

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The data was qualified as estimated (UJ) due to recovery problems with ICV (>110%). The LCS was not digested; therefore, it was not indicative of system control.

NOTES: The AA metals analyses were performed using procedure LA-355-131.

- Refer to the corresponding attachments for explanations of any problems.

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name J. M. Jones

Date 1-5-94

QC Check: HOLDING TIMES

COMMENTS: Analytical holding times were assessed to determine whether the requirements for GHAA analyses were met. The maximum allowable holding time for metals evaluated by GHAA is six months. The samples were collected on 8-1-93 and acquired by the 222-S lab on 8-2-93. The samples were received in good condition and preserved in accordance with SW-846.

ACTION: No action was required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-180

ASD  
1-14-94  
174

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: INSTRUMENT CALIBRATION

**COMMENTS:** The data was examined to determine whether the instruments used were calibrated at the correct frequency and that the calibration was performed correctly. All instruments must be calibrated on a daily basis or upon each set-up. Atomic Absorption calibration must be performed with a minimum of a blank and 3 standards with a minimum correlation coefficient of 0.995. Data is qualified as unusable if reported from an instrument that was not calibrated or was calibrated with less than the minimum number of standards. Associated sample results were qualified as estimated if the correlation coefficient is less than 0.995.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-181

000175

WHC-SD-WM-DP-053  
ADDENDUM 14 REV. 0

INORGANIC QC

Name: J. M. Jones

Date: 1-5-94

QC Check: INITIAL AND CONTINUING CALIBRATION VERIFICATION

COMMENTS: An Initial Calibration Verification (ICV) standard must be run at the beginning of each run. A Continuing Calibration Standard (CCV) must be run at a 10% frequency. The recoveries for all ICVs must be within  $\pm 10\%$  of the true value and the recoveries for CCVs within  $\pm 20\%$  for AA and  $\pm 10\%$  for ICP. If the ICV/CCV results are outside the acceptable range, all associated sample results are qualified as estimated.

ACTION: The ICV digested standard was outside the 90-110% criteria range, and there was no ending CCV. The LCS was utilized as an alternative, but it was not digested and, therefore, not indicative of system control. The results were qualified according to the following table.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V21	As	<0.013 ug/ml UJ
	As re-run #1	<0.013 UJ
	As re-run #2	<0.013 UJ
	Se	<0.013 UJ
V23	As	<0.013 ug/ml UJ
	As re-run #1	<0.013 UJ
	As re-run #2	<0.013 UJ
	Se	<0.013 UJ
V26	As	<0.013 ug/ml UJ
	Se	<0.013 UJ
V27	As	<0.013 ug/ml UJ
	Se	<0.013 UJ
V28	As	<0.013 ug/ml UJ
	Se	<0.013 UJ
V31	As	<0.013 ug/ml UJ
	Se	<0.013 UJ

1A-182

QSD  
1-14-94

176

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: BLANKS

**COMMENTS:** Calibration and preparation blanks were evaluated for the presence of contaminants. Calibration blanks should be run at a 10% frequency. At least one preparation blank is required for each sample batch. If the concentration of analytes in the sample exceeded 5 times the blank concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** The ICB and Prep Blank were one in the same.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-183

000177

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: INTERFERENCE CHECK SAMPLE

**COMMENTS:** The ICP Interference Check Sample (ICS) is run to verify the instrumental interelement and background correction factors. An ICS must be run at the beginning and end of each sample analysis run or twice per 8 hour shift. The results for the ICS solution AB analysis must fall within the control limits of  $\pm 20\%$  of the true value. In addition, the ICS raw data is examined for results with an absolute value of  $> IDL$  for those analytes which are not present in the ICS solution. Associated sample results are qualified as estimated when the ICS criteria are not met.

**ACTION:** Not applicable for As/Se.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name: J. M. Jones

Date: 1-5-94

QC Check: LABORATORY CONTROL STANDARD

**COMMENTS:** The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. All LCS results must fall within the control limits of  $\pm 20\%$  of the true value. If the LCS recovery is  $> 120\%$  or  $50 - 79\%$ , sample results are qualified as estimated. Results associated with an LCS recovery of  $< 50\%$  are qualified as unusable.

**ACTION:** The LCS was not digested, but the samples were digested. This cannot be used for system control. The results were qualified as indicated in the following table.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V21	As	<0.013 ug/ml UJ
	As re-run #1	<0.013 UJ
	As re-run #2	<0.013 UJ
	Se	<0.013 UJ
V23	As	<0.013 ug/ml UJ
	As re-run #1	<0.013 UJ
	As re-run #2	<0.013 UJ
	Se	<0.013 UJ
V26	As	<0.013 ug/ml UJ
	Se	<0.013 UJ
V27	As	<0.013 ug/ml UJ
	Se	<0.013 UJ
V28	As	<0.013 ug/ml UJ
	Se	<0.013 UJ
V31	As	<0.013 ug/ml UJ
	Se	<0.013 UJ

*JJD*  
1-14-94

1A-185

179

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are indicators of laboratory precision based on each sample matrix. Duplicate analysis must be performed at a 5% frequency or 1 per batch, whichever is greater. The relative percent deviation (RPD) for duplicate analyses should be less than 20% for sample results greater than 10 times the IDL. If the RPD is greater than 20%, the associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-186

000180

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: MATRIX SPIKE/MATRIX SPIKE DUPLICATE

**COMMENTS:** Matrix spikes sample analysis provide information about the effect of each sample matrix on the digestion and measurement methodology. Matrix spikes must be performed at a 20% frequency and recoveries should be between 75-125%. If the spike result is between 30-74% or >125%, results are qualified as estimated. Sample results associated with a spike recovery of less than 30% are qualified as unusable.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-187

000181

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: CONTRACT REQUIRED DETECTION LIMIT STANDARD

**COMMENTS:** A Contract Required Detection Limit Standard (CRA) is performed to evaluate instrument performance near the detection limit for AA and ICP metals. The control limit is only advisory.

**ACTION:** Standard was not run for As/Se.

sample #      constituent      value/qualifier

1A-188

182  
~~000183~~  
JR 1/13/94

INORGANIC QC

Name J.M. JONES

Date 1-5-94

QC Check: SERIAL DILUTION

**COMMENTS:** Serial dilutions are run to determine whether significant physical or chemical interferences exist due to sample matrix. In addition, the results of the serial dilution can be used to determine whether sample results greater than the instrument linear range can be reported as valid results. Analyte results for a five fold dilution that are greater than 50 times the IDL must agree within 10% difference (%D) of the original results. If the criteria are not met, the results are qualified as estimated. In the presence of negative interferences, professional judgement is used to qualify the data.

**ACTION:** Not applicable for As/Se.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-189

183

~~000182~~  
1/12/94



WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC OC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: HOLDING TIMES

**COMMENTS:** Analytical holding times were assessed to determine whether the requirements for metals analyses were met. The maximum holding times for mercury is 13 days if stored in plastic and 38 days if stored in glass. The samples were collected on 8-1-93 and received by the 222-S Laboratory on 8-2-93. All samples were received in good condition and preserved in accordance with SW-846.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-191

000185

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: INSTRUMENT CALIBRATION

**COMMENTS:** The data was examined to determine whether the instruments used were calibrated at the correct frequency and that the calibration was performed correctly. All instruments must be calibrated on a daily basis or upon each set-up. Atomic Absorption calibration must be performed with a minimum of a blank and 3 standards with a minimum correlation coefficient of 0.995. Data is qualified as unusable if reported from an instrument that was not calibrated or was calibrated with less than the minimum number of standards. Associated sample results were qualified as estimated if the correlation coefficient is less than 0.995.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 192

000186

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: INITIAL AND CONTINUING CALIBRATION VERIFICATION

**COMMENTS:** An Initial Calibration Verification (ICV) standard must be run at the beginning of each run. A Continuing Calibration Standard (CCV) must be run at a 10% frequency. The recoveries for all ICVs must be within  $\pm 10\%$  of the true value and the recoveries for CCVs within  $\pm 20\%$  for AA and  $\pm 10\%$  for ICP. If the ICV/CCV results are outside the acceptable range, all associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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JA 193

000187

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: BLANKS

**COMMENTS:** Calibration and preparation blanks were evaluated for the presence of contaminants. Calibration blanks should be run at a 10% frequency. At least one preparation blank is required for each sample batch. If the concentration of analytes in the sample exceeded 5 times the blank concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-194

000188

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: INTERFERENCE CHECK SAMPLE

**COMMENTS:** The ICP Interference Check Sample (ICS) is run to verify the instrumental interelement and background correction factors. An ICS must be run at the beginning and end of each sample analysis run or twice per 8 hour shift. The results for the ICS solution AB analysis must fall within the control limits of  $\pm 20\%$  of the true value. In addition, the ICS raw data is examined for results with an absolute value of  $> IDL$  for those analytes which are not present in the ICS solution. Associated sample results are qualified as estimated when the ICS criteria are not met.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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LA-195

000189

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rollosson

Date 1-3-94

QC Check: LABORATORY CONTROL STANDARD

**COMMENTS:** The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. All LCS results must fall within the control limits of  $\pm 20\%$  of the true value. If the LCS recovery is  $> 120\%$  or  $50 - 79\%$ , sample results are qualified as estimated. Results associated with an LCS recovery of  $< 50\%$  are qualified as unusable.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-196

000150

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are indicators of laboratory precision based on each sample matrix. Duplicate analysis must be performed at a 5% frequency or 1 per batch, whichever is greater. The relative percent deviation (RPD) for duplicate analyses should be less than 20% for sample results greater than 10 times the IDL. If the RPD is greater than 20%, the associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-197

000191

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
INORGANIC QC

Name M.I. Weyns-Rolloson

Date 1-3-94

QC Check: MATRIX SPIKE/MATRIX SPIKE DUPLICATE

**COMMENTS:** Matrix spikes sample analysis provide information about the effect of each sample matrix on the digestion and measurement methodology. Matrix spikes must be performed at a 20% frequency and recoveries should be between 75-125%. If the spike result is between 30-74% or >125%, results are qualified as estimated. Sample results associated with a spike recovery of less than 30% are qualified as unusable.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-198

000192

ICP DATA ASSESSMENT

DATE	<u>01-05-94</u>	SAMPLES/MATRIX	<u>V21-8750/WATER</u>
REVIEWED BY	<u>M.I. Weyns-Rollosson</u> <i>dw 1/6/94</i>		<u>V23-8750/WATER</u>
LABORATORY	<u>222-S</u>		<u>V23R-8750/WATER</u>
CASE #	<u>242 EVAPORATOR</u>		<u>V26-8750/WATER</u>
SDG #	<u>241-AP-107</u>		<u>V27-8750/WATER</u>
			<u>V28-8750/WATER</u>
			<u>V31-8750/WATER</u>

DATA ASSESSMENT SUMMARY

	<u>ICP</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
1. <u>Chain of Custody/Holding Times</u>	<u>0</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
2. <u>Instrument Calibration</u>	<u>0</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
3. <u>ICV/CCV Standards</u>	<u>0</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
4. <u>Blanks</u>	<u>X</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
5. <u>Interference Check Sample</u>	<u>0</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
6. <u>Laboratory Control Sample</u>	<u>X</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
7. <u>Duplicate Analysis</u>	<u>X</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
8. <u>Matrix Spike/Matrix Spike Dup.</u>	<u>0</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
9. <u>CRDL Standard</u>	<u>N/A</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
10. <u>Serial Dilution</u>	<u>N/A</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>
11. <u>Other Quality Control</u>	<u>N/A</u>	<u>_____</u>	<u>_____</u>	<u>_____</u>

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The ICP metal analyses were conducted in accordance with the recommended quality control requirements, with a few exceptions. Several results were qualified as estimated for exceeding control limits for duplicates, laboratory control samples, and blanks.

NOTES: The ICP metals analyses were performed using procedure LA-505-151.

- Refer to the corresponding attachments for explanations of any problems.

*1A-159*

000153

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: HOLDING TIMES

**COMMENTS:** Analytical holding times were assessed to determine whether the requirements for metals analyses were met. The maximum holding time for ICP metals is 180 days. The samples were collected on 08-01-93 and received by the 222-S Laboratory on 08-02-93. All samples were received in good condition and preserved in accordance with SW-846.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 200

000194

WHC-SD-WM-DP-053  
ADDENDUM 14 REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: INSTRUMENT CALIBRATION

**COMMENTS:** The data was examined to determine whether the instruments used were calibrated at the correct frequency and that the calibration was performed correctly. All instruments must be calibrated on a daily basis or upon each set-up. Atomic Absorption calibration must be performed with a minimum of a blank and 3 standards with a minimum correlation coefficient of 0.995. Data is qualified as unusable if reported from an instrument that was not calibrated or was calibrated with less than the minimum number of standards. Associated sample results were qualified as estimated if the correlation coefficient is less than 0.995.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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14-201

000195

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: INITIAL AND CONTINUING CALIBRATION VERIFICATION

**COMMENTS:** An Initial Calibration Verification (ICV) standard must be run at the beginning of each run. A Continuing Calibration Standard (CCV) must be run at a 10% frequency. The recoveries for all ICVs must be within  $\pm 10\%$  of the true value and the recoveries for CCVs within  $\pm 20\%$  for AA and  $\pm 10\%$  for ICP. If the ICV/CCV results are outside the acceptable range, all associated sample results are qualified as estimated.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 202

000196

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: BLANKS

**COMMENTS:** Calibration and preparation blanks were evaluated for the presence of contaminants. Calibration blanks should be run at a 10% frequency. At least one preparation blank is required for each sample batch. If the concentration of analytes in the sample exceeded 5 times the blank concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** The following analytes were qualified as non-detect since the sample results were not five times the amount in the preparation blank: Iron in samples V21, V23, and V23R; Magnesium in samples V23R and V26; Calcium in samples V26 and V27; and Zinc in sample V26. The silver result for sample V23R was qualified as non-detect because the sample result was not five times the amount in the continuing calibration blank. The iron result in sample V21 was qualified as estimated since the absolute value of the blank, which was negative, was not greater than two times the IDL.

<u>sample #</u>	<u>constituent</u>	<u>value(ug/L)/qualifier</u>	
V21	Fe	11.0	UJ
V23	Fe	11.0	U
V23R	Ag	6.0	U
	Fe	21.0	U
	Mg	5.0	U
V26	Mg	3.0	U
	Ca	8.0	U
	Zn	3.0	U
V27	Ca	8.0	U

1A-203

GG0197

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rollosson

Date 01-05-94

QC Check: INTERFERENCE CHECK SAMPLE

**COMMENTS:** The ICP Interference Check Sample (ICS) is run to verify the instrumental interelement and background correction factors. An ICS must be run at the beginning and end of each sample analysis run or twice per 8 hour shift. The results for the ICS solution AB analysis must fall within the control limits of  $\pm 20\%$  of the true value. In addition, the ICS raw data is examined for results with an absolute value of  $> IDL$  for those analytes which are not present in the ICS solution. Associated sample results are qualified as estimated when the ICS criteria are not met.

**ACTION:** All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-204

000158

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: LABORATORY CONTROL STANDARD

**COMMENTS:** The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of all steps in the analysis, including sample preparation. All LCS results must fall within the control limits of  $\pm 20\%$  of the true value. If the LCS recovery is  $> 120\%$  or  $50 - 79\%$ , sample results are qualified as estimated. Results associated with an LCS recovery of  $< 50\%$  are qualified as unusable.

**ACTION:** The ICP sample results were qualified as indicated by the following table.

<u>sample #</u>	<u>constituent</u>	<u>value(ug/L)/qualifier</u>	
V21	Ca	20500	J
	Mg	875	J
	Zn	560	J
V23	Ca	47100	J
	Mg	1990	J
	Zn	2100	J
V23R	Na	2,260,000	J
V26	Ca	8.0	UJ
	Ag	50.9	J
V27	Ca	8.0	UJ
	Ag	50.0	J
V28	Ca	113000	J
	Ag	51.8	J
V31	Ag	30.0	UJ

1A-205

000199

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are indicators of laboratory precision based on each sample matrix. Duplicate analysis must be performed at a 5% frequency or 1 per batch, whichever is greater. The relative percent deviation (RPD) for duplicate analyses should be less than 20% for sample results greater than 10 times the IDL. If the RPD is greater than 20%, the associated sample results are qualified as estimated.

**ACTION:** The ICP results were qualified as indicated by the following table.

<u>sample #</u>	<u>constituent</u>	<u>value(ug/L)/qualifier</u>	
V21	Ca	20500	J
	Cr	30.8	J
	Fe	11.0	UJ
	Pb	190	UJ
	Zn	560	J
V23	Ba	16.2	J
	Ca	47100	J
	Cd	20.0	UJ
	Cr	30.0	UJ
	Fe	11.0	UJ
	Mg	1990	J
V23R	Ca	2590	J
	Fe	21.0	UJ
V26	Ca	8.0	UJ
	Pb	232	J
	Mg	3.0	UJ
	Mn	15.0	UJ
	Zn	3.0	UJ
V27	Ag	50.0	J
	Al	1290	J
	Ba	15.8	J
	Mn	15.0	UJ
	Zn	234	J

1A-206

GG0200

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: DUPLICATE ANALYSIS CONTINUED

<u>sample #</u>	<u>constituent</u>	<u>value(ug/L)/qualifier</u>	
V28	Al	1260	J
	Ba	52.4	J
	Ca	113000	J
	Fe	399	J
	Mg	6900	J
	Mn	189	J
	Zn	5020	J
V31	Ag	30.0	UJ
	Al	895	J
	Ba	15.0	UJ
	Ca	3530	J
	Fe	272	J
	Mg	375	J
	Mn	15.0	UJ
	Zn	382	J

/A-207

000201

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

INORGANIC QC

Name M.I. Weyns-Rolloson

Date 01-05-94

QC Check: MATRIX SPIKE/MATRIX SPIKE DUPLICATE

COMMENTS: Matrix spikes sample analysis provide information about the effect of each sample matrix on the digestion and measurement methodology. Matrix spikes must be performed at a 20% frequency and recoveries should be between 75-125%. If the spike result is between 30-74% or >125%, results are qualified as estimated. Sample results associated with a spike recovery of less than 30% are qualified as unusable.

ACTION: All criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-208

000202

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1A-209

000203

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1A 211

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1A-212

G00206

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WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

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1A-214

000208

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**WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
RADIOCHEMICAL DATA ASSESSMENT**

DATE	<u>01-06-94</u>	SAMPLES/MATRIX	<u>V31-8730,8725</u>
REVIEWED BY	<u>D.E. STROUP</u> <i>Dec 1-7-94</i>		<u>V34-8730,8725</u>
LABORATORY	<u>222-S</u>		<u>WATER</u>
CASE #	<u>242A EVAPORATOR</u>		
SDG #	<u>107-AP-A-222-088</u>		

**DATA ASSESSMENT SUMMARY**

	<u>GEA</u>	<u>A&amp;B</u>	<u>      </u>	<u>      </u>
1. <u>Chain of Custody</u>	<u>0</u>	<u>0</u>	<u>      </u>	<u>      </u>
2. <u>Initial Calibration</u>	<u>0</u>	<u>0</u>	<u>      </u>	<u>      </u>
3. <u>Efficiency Checks</u>	<u>0</u>	<u>0</u>	<u>      </u>	<u>      </u>
4. <u>Background Checks</u>	<u>0</u>	<u>0</u>	<u>      </u>	<u>      </u>
5. <u>Preparation Blanks</u>	<u>X</u>	<u>0</u>	<u>      </u>	<u>      </u>
6. <u>MS/Tracers/Carriers</u>	<u>0</u>	<u>X</u>	<u>      </u>	<u>      </u>
7. <u>Duplicate Analysis</u>	<u>0</u>	<u>0</u>	<u>      </u>	<u>      </u>
8. <u>LCS</u>	<u>0</u>	<u>0</u>	<u>      </u>	<u>      </u>

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The data is acceptable with the minor qualifications noted above and on the corresponding attachments.

NOTES: V31 is the field blank and V34 is a composite sample.

o Refer to the corresponding attachments for explanation of any problems.

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-94

QC Check: CHAIN OF CUSTODY

COMMENTS: The samples were collected 8-1-93 and analyzed with the required 180 days

ACTION: No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A218

000212

WHC-SD-WM-DP-053  
ADDENDUM 14 REV. 0

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-93

QC Check: INITIAL CALIBRATION

**COMMENTS:** The data packages are reviewed to verify that the instrument was calibrated within the time period specified by the laboratory standard operating procedure or manufacturer's instruction. Instrument efficiencies are determined from the initial calibration. If the instrument was not calibrated within the specified time period, all associated results are qualified as unusable.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-219

000213

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-94

QC Check: EFFICIENCY CHECKS

**COMMENTS:** Efficiency checks are counted to ensure that acceptable instrument performance is maintained on a day to day basis. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (< 3 sigma). If efficiency QC criteria are not met, sample results are qualified as estimated.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-220

000214

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-93

QC Check: BACKGROUND CHECKS

**COMMENTS:** Background radiation is measured by counting a simulated sample or source which identical to the actual sample except for the absence of radioactivity from a sample source. Background checks must be acquired for each detector system on a regular basis. The frequency of background checks is dependent on the sample count time.

<u>Count Time</u>	<u>Background Frequency</u>
0-1 hour	1 per 8 hours
1-8 hours	1 per 24 hours
>8 hours	1 per week

Background checks should not deviate more than 3 times the standard deviation of normal operating conditions. If the background results are outside of the specified frequency or control limits, sample results are qualified as estimated.

**ACTION:** All criteria were met.

sample #      constituent      value/qualifier

1A 221

000215

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-94

QC Check: PREPARATION BLANKS

**COMMENTS:** Preparation blanks were evaluated for the presence of contaminants. At least one preparation blank is required for each sample batch. If the concentration of analytes in the blanks exceeded 5 times the sample concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

There is possible Cs-137 contamination of the GEA sample. A re-run was completed.

**ACTION:** Qualify the original GEA V34 Cs-137 results as estimated (J).

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V34	Cs-137	1.23E-4 uCi/mL J

1A-222

000216

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-94

QC Check: MATRIX SPIKES/TRACERS/CARRIERS

**COMMENTS:** Matrix spikes, tracers, and carriers are used in radiochemical analyses to indicate overall accuracy for a given matrix. Matrix spikes are not required for GEA or Total (Gross) Beta analyses. The control limits for matrix spikes, carriers, or tracers is less than three standard deviations of normal operating conditions. Results outside these limits are qualified as estimated or unusable based on the judgement of the reviewer. The Total Alpha spike and spike duplicate recoveries were low and a re-run was completed. The re-run spike recovery was low and the duplicate spike recovery was within limits. The chemist states in the narrative that the re-run spike was not prepared properly. In a cc: mail correspondence it is stated that solids were noted on the counting planchet after sample evaporation and the conclusion was made that self adsorption was the cause for the low recoveries on the initial and re-run.

**ACTION:** Qualify the Total Alpha results as estimated, non detect (UJ).

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>	
V31	Total Alpha	<1.21E-6 uCi/mL	UJ
V34	" "	<1.96E-6 "	UJ
V34 rerun	" "	<7.14E-7 "	UJ

1A-223

000217

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analysis are performed to monitor the precision of the method. Duplicate results should be within 3 sigma of normal operating conditions. If either the sample or duplicate is below the Minimum Detectable Activity (MDA) then no control limit applies. All results outside the control limit are qualified as estimated.

**ACTION:** All QC criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 224

000218

RADIOCHEMICAL QC

Name D.E. Stroup

Date 01-06-94

QC Check: LABORATORY CONTROL SAMPLES

**COMMENTS:** The Laboratory Control Sample (LCS) is a monitor of the overall performance of analytical method, including sample preparation. An LCS must be analyzed with each batch. The LCS recoveries must be within 3 times the standard deviation of normal operating conditions. Results outside these limits are qualified as estimated or unusable depending on the mood of the validator.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-225

000219

RADIOCHEMICAL DATA ASSESSMENT

DATE	<u>01-06-94</u>	SAMPLES/MATRIX	<u>V34-5787/WATER</u>
REVIEWED BY	<u>M.I. Weyns-Rollosson</u> <i>dw 1/6/94</i>		<u>V34R-5787/WATER</u>
LABORATORY	<u>222-S</u>		<u>V34-5788/WATER</u>
CASE #	<u>242 EVAPORATOR</u>		<u>V34-8789/WATER</u>
SDG #	<u>241-AP-107</u>		<u>V34-8784/WATER</u>

DATA ASSESSMENT SUMMARY

LIQUID SCINTILLATION

	<u>H-3</u>	<u>C-14</u>	<u>Se-79</u>	<u>Tc-99</u>
1. <u>Chain of Custody</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
2. <u>Initial Calibration</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
3. <u>Efficiency Checks</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
4. <u>Background Checks</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
5. <u>Preparation Blanks</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
6. <u>MS/Tracers/Carriers</u>	<u>M</u>	<u>0</u>	<u>M</u>	<u>0</u>
7. <u>Duplicate Analysis</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
8. <u>LCS</u>	<u>0</u>	<u>0</u>	<u>X</u>	<u>0</u>

0 = data had no problems

X = minor problems, data may be qualified

M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The liquid scintillation analyses were conducted in accordance with the recommended quality control requirements with a minor qualification poor LCS recoveries during the Selenium-79 analysis and major qualifications due to poor matrix spike/carrier recoveries for the analysis of Tritium and Selenium-79.

NOTES: The liquid scintillation analyses were performed using the following procedures: Tritium (LA-218-114), Carbon-14 (LA-348-104), Selenium-79 (LA-365-132), and Technetium-99 (LA-438-101).

o Refer to the corresponding attachments for explanation of any problems.

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 11-23-93

QC Check: CHAIN OF CUSTODY

**COMMENTS:** The sample was collected by WHC on 08-01-93 and transferred in chilled containers without incident to the 222-S Laboratory for analysis. The 222-S Laboratory received the samples on 08-02-93 and analysis took place between 8-17-93 and 10-15-93, within the 180 day holding time specifications.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-227

000221

RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 01-06-94

QC Check: INITIAL CALIBRATION

**COMMENTS:** The data packages are reviewed to verify that the instrument was calibrated within the time period specified by the laboratory standard operating procedure or manufacturer's instruction. Instrument efficiencies are determined from the initial calibration. If the instrument was not calibrated within the specified time period, all associated results are qualified as unusable.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-228

000222

WHC-SD-WM-DP-053  
ADDENDUM 1/ REV. 0

RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 01-06-94

QC Check: EFFICIENCY CHECKS

**COMMENTS:** Efficiency checks are counted to ensure that acceptable instrument performance is maintained on a day to day basis. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (< 3 sigma). If efficiency QC criteria are not met, sample results are qualified as estimated.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-229

000223

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name M.I. Weyns-Rollosson

Date 01-06-94

QC Check: BACKGROUND CHECKS

**COMMENTS:** Background radiation is measured by counting a simulated sample or source which is identical to the actual sample except for the absence of radioactivity from a sample source. Background checks must be acquired for each detector system on a regular basis. The frequency of background checks is dependent on the sample count time.

<u>Count Time</u>	<u>Background Frequency</u>
0-1 hour	1 per 8 hours
1-8 hours	1 per 24 hours
>8 hours	1 per week

Background checks should not deviate more than 3 times the standard deviation of normal operating conditions. If the background results are outside of the specified frequency or control limits, sample results are qualified as estimated.

**ACTION:** No action is required.

sample #      constituent      value/qualifier

1A-230

000224

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 01-06-94

QC Check: PREPARATION BLANKS

**COMMENTS:** Preparation blanks were evaluated for the presence of contaminants. At least one preparation blank is required for each sample batch. If the concentration of analytes in the blanks exceeded 5 times the sample concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-231

000225

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 01-06-94

QC Check: MATRIX SPIKES/TRACERS/CARRIERS

**COMMENTS:** Matrix spikes, tracers, and carriers are used in radiochemical analyses to indicate overall accuracy for a given matrix. Matrix spikes are not required for GEA or Total (Gross) Beta analyses. The control limit for matrix spikes, carriers, or tracers is less than three standard deviations of normal operating conditions. Results outside these limits are qualified as estimated or unusable based on the judgement of the reviewer.

**ACTION:** The matrix spike recoveries for the tritium analysis of sample V34 and its duplicate were extremely high (3438% and 2989%) and the carrier recoveries for Selenium-79 were extremely low (23% and 28.5%), resulting in the qualification of these results as unusable. The tritium analysis of sample V34 was re-run because the spike was prepared incorrectly. The matrix spike recovery of the re-analysis was acceptable.

<u>sample #</u>	<u>constituent</u>	<u>value (uCi/ml)/qualifier</u>	
V34	H-3	3.35E-2	R
	Se-79	<5.38E-6	R

1A-232

000226

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 01-06-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are performed to monitor the precision of the method. Duplicate results should be within 3 sigma of normal operating conditions. If either the sample or duplicate is below the Minimum Detectable Activity (MDA) then no control limit applies. All results outside the control limit are qualified as estimated.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-233

000227

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name M.I. Weyns-Rolloson

Date 01-06-94

QC Check: LABORATORY CONTROL SAMPLES

**COMMENTS:** The Laboratory Control Sample (LCS) is a monitor of the overall performance of the analytical method, including sample preparation. An LCS must be analyzed with each batch. The LCS recoveries must be within 3 times the standard deviation of normal operating conditions. Results outside these limits are qualified as estimated or unusable depending on the judgement of the validator.

**ACTION:** An LCS was not run with the Se-79 analysis of sample V34, resulting in the qualification of this result as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V34	Se-79	<5.38E-6uCi/ml UJ

1A 234

000228

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL DATA ASSESSMENT

DATE	<u>January 7, 1993</u>	SAMPLES/MATRIX	<u>V-34/Liquid</u>
REVIEWED BY	<u>D. J. Smith</u>	<i>[Signature]</i> <u>1/7/93</u>	_____
LABORATORY	<u>222-S</u>		_____
CASE #	<u>241-AP-107</u>		_____
SDG #	<u>241-AP-107</u>		_____

DATA ASSESSMENT SUMMARY

	<u>Sr-90</u>	_____	_____	_____
1. <u>Chain of Custody</u>	<u>0</u>	_____	_____	_____
2. <u>Requested/Reported Anal.</u>	<u>0</u>	_____	_____	_____
3. <u>Holding Times</u>	<u>0</u>	_____	_____	_____
4. <u>Calibration</u>	<u>0</u>	_____	_____	_____
5. <u>Efficiency Checks</u>	<u>X</u>	_____	_____	_____
6. <u>Background Checks</u>	<u>X</u>	_____	_____	_____
7. <u>Duplicate Analysis</u>	<u>0</u>	_____	_____	_____
8. <u>MS/Tracers/Carriers</u>	<u>0</u>	_____	_____	_____
9. <u>Analytical Blanks</u>	<u>X</u>	_____	_____	_____
10. <u>LCS</u>	<u>0</u>	_____	_____	_____
11. <u>Other QC Checks</u>	<u>0</u>	_____	_____	_____

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The overall quality of the data is good. All results were qualified as estimated for missing efficiency and background check documentation. The Sr-90 results were also qualified for preparation blank contamination above acceptable levels.

NOTES: \_\_\_\_\_

o Refer to the corresponding attachments for explanation of any problems.

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: CHAIN OF CUSTODY

**COMMENTS:** The sample was collected by WHC on 8/1/93 and transferred in without incident to the 222S laboratory. The laboratory received the samples in good condition. All chain of custody documentation has been included in the data package.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-236

000230

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: REQUESTED VERSUS REPORTED ANALYSES

**COMMENTS:** The Chain of Custody and Sample Analysis Request forms were compared with the analysis reported by the laboratory. All analysis requested performed according to instruction.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 237

231

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check HOLDING TIMES

**COMMENTS:** Samples should be analyzed within the period of 180 days from the date of sampling. Samples should be properly contained and preserved (e.g., acidified) in accordance with laboratory standard procedures, to ensure that sample integrity is maintained. Holding times for each radionuclide were established by comparing the sampling date on the chain-of-custody record with the dates of analysis found in the data package.

Analysis date - sample date = Radionuclide holding time

All applicable holding times were met.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-238

000232

WHC-SD-WM-DP-053  
ADDENDUM 1<sup>A</sup> REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: INITIAL CALIBRATION

**COMMENTS:** The data packages are reviewed to verify that the instrument was calibrated within the time period specified by the laboratory standard operating procedure or manufacturer's instruction. Instrument efficiencies are determined from the initial calibration. If the instrument was not calibrated within the specified time period, all associated results are qualified as unusable.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-239

000233

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: EFFICIENCY CHECKS

**COMMENTS:** Efficiency checks are counted to ensure that acceptable instrument performance is maintained on a day to day basis. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (< 3 sigma). If efficiency QC criteria are not met, sample results are qualified as estimated.

Since efficiency check data was not provided, all results are qualified as estimated.

**ACTION:** Qualify Sr-90 as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-34	Sr-90	1.14 E-4 uCi/ml J

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: BACKGROUND CHECKS

**COMMENTS:** Background radiation is measured by counting a simulated sample or source which identical to the actual sample except for the absence of radioactivity from a sample source. Background checks must be acquired for each detector system on a regular basis. The frequency of background checks is dependent on the sample count time.

<u>Count Time</u>	<u>Background Frequency</u>
0-1 hour	1 per 8 hours
1-8 hours	1 per 24 hours
>8 hours	1 per week

Background checks should not deviate more than 3 times the standard deviation of normal operating conditions. If the background results are outside of the specified frequency or control limits, sample results are qualified as estimated.

Since background checks were not provided, all Sr-90 results are qualified as estimated.

**ACTION:** Qualify Sr-90 results as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-34	Sr-90	1.14 E-4 uCi/ml J

1A-241

000235

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analysis must be performed with every analytical batch or every twenty samples, whichever is more frequent. This requirement may be satisfied with the analysis of an MS/MSD sample. Method or program DQO specified control limits shall be applied to sample results where they exist, otherwise the Relative Percent Difference (RPD) shall be less than 20% for water samples (<35% for soils) if the sample result is greater than 5X times the RDL. If the sample result is less than 5 times the RDL, the difference between the primary and duplicate results must be less than the RDL for water samples and less than 2 times the RDL for soils. If both sample and duplicate results are below the Method Detection Limit (MDL) or Sample Quantitation Limit (SQL), then no control limit applies.

**ACTION:** All QC criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-242

236

~~000238~~  
AQ 1/13/93

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: MATRIX SPIKES/TRACERS/CARRIERS

**COMMENTS:** Matrix spikes, tracers, and carriers are used in radiochemical analyses to indicate overall accuracy for a given matrix. Matrix spikes are not required for GEA or Total (Gross) Beta analyses. The control limits for matrix spikes, carriers, or tracers is less than three standard deviations of normal operating conditions. Results outside these limits are qualified as estimated or unusable based on the judgement of the reviewer.

**ACTION:** All carrier recoveries were acceptable.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-243

000237

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: PREPARATION BLANKS

**COMMENTS:** Preparation blanks were evaluated for the presence of contaminants. At least one preparation blank is required for each sample batch. If the concentration of analytes in the blanks exceeded 20% of the sample concentration, the associated sample results were qualified as estimated. If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated. In this case, the Sr-90 blank result was 30% of the sample result.

**ACTION:** Qualify all Sr-90 results as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-34	Sr-90	1.14 E-4 uCi/ml J

1A-244

238  
~~000236~~  
1/13/94

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: LABORATORY CONTROL SAMPLES

**COMMENTS:** The Laboratory Control Sample (LCS) is a monitor of the overall performance of analytical method, including sample preparation. An LCS must be analyzed with each batch. The LCS recoveries must be within 3 times the standard deviation of normal operating conditions. Results outside these limits are qualified as estimated or unusable depending on the judgement of the validator.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-245

000239

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: OTHER QUALITY CONTROL

**COMMENTS:** The radiochemical data was examined for compliance with specific project Data Quality Objectives and the Statement of Work. Any trends observed in the performance of an instrument, method, or laboratory of the course of the analysis are noted.

**ACTION:** All analyses were performed according to the requirements of the QAPP and TPP. No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-246

000240

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL DATA ASSESSMENT

DATE January 7, 1993 SAMPLES/MATRIX V-34/Liquid  
 REVIEWED BY D. J. Smith *[Signature]* 1/7/93  
 LABORATORY 222-S  
 CASE # 241-AP-107  
 SDG # 241-AP-107

DATA ASSESSMENT SUMMARY

	I-129			
1. <u>Chain of Custody</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
2. <u>Requested/Reported Anal.</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
3. <u>Holding Times</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
4. <u>Calibration</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
5. <u>Efficiency Checks</u>	<u>X</u>	<u>    </u>	<u>    </u>	<u>    </u>
6. <u>Background Checks</u>	<u>X</u>	<u>    </u>	<u>    </u>	<u>    </u>
7. <u>Duplicate Analysis</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
8. <u>MS/Tracers/Carriers</u>	<u>NA</u>	<u>    </u>	<u>    </u>	<u>    </u>
9. <u>Analytical Blanks</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
10. <u>LCS</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>
11. <u>Other QC Checks</u>	<u>0</u>	<u>    </u>	<u>    </u>	<u>    </u>

0 = data had no problems  
 X = minor problems, data may be qualified  
 M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The overall quality of the data is good. All results were qualified as estimated for missing efficiency and background check documentation.

NOTES: \_\_\_\_\_

o Refer to the corresponding attachments for explanation of any problems.

*1A 247*

000241

WHC-SD-WM-DP-053  
ADDENDUM 1/REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: CHAIN OF CUSTODY

COMMENTS: The sample was collected by WHC on 8/1/93 and transferred in without incident to the 222S laboratory. The laboratory received the samples in good condition. All chain of custody documentation has been included in the data package.

ACTION: No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-248

000242

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: REQUESTED VERSUS REPORTED ANALYSES

COMMENTS: The Chain of Custody and Sample Analysis Request forms were compared with the analysis reported by the laboratory. All analysis requested performed according to instruction.

ACTION: No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A 249

243

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check HOLDING TIMES

COMMENTS: Samples should be analyzed within the period of 180 days from the date of sampling. Samples should be properly contained and preserved (e.g., acidified) in accordance with laboratory standard procedures, to ensure that sample integrity is maintained. Holding times for each radionuclide were established by comparing the sampling date on the chain-of-custody record with the dates of analysis found in the data package.

Analysis date - sample date = Radionuclide holding time

All applicable holding times were met.

ACTION: No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-250

000244

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0  
RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: INITIAL CALIBRATION

**COMMENTS:** The data packages are reviewed to verify that the instrument was calibrated within the time period specified by the laboratory standard operating procedure or manufacturer's instruction. Instrument efficiencies are determined from the initial calibration. If the instrument was not calibrated within the specified time period, all associated results are qualified as unusable.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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(A) 251

000245

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: EFFICIENCY CHECKS

COMMENTS: Efficiency checks are counted to ensure that acceptable instrument performance is maintained on a day to day basis. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (< 3 sigma). If efficiency QC criteria are not met, sample results are qualified as estimated.

Since efficiency check data was not provided, all results are qualified as estimated.

ACTION: Qualify I-129 as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-34	I-129	<4.35 E-5 uCi/ml UJ

1A-252

000246

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: BACKGROUND CHECKS

**COMMENTS:** Background radiation is measured by counting a simulated sample or source which identical to the actual sample except for the absence of radioactivity from a sample source. Background checks must be acquired for each detector system on a regular basis. The frequency of background checks is dependent on the sample count time.

Count Time

Background Frequency

0-1 hour  
1-8 hours  
>8 hours

1 per 8 hours  
1 per 24 hours  
1 per week

Background checks should not deviate more than 3 times the standard deviation of normal operating conditions. If the background results are outside of the specified frequency or control limits, sample results are qualified as estimated.

Since background checks were not provided, all I-129 results are qualified as estimated.

**ACTION:** Qualify I-129 results as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V-34	I-129	<4.35 E-4 uCi/ml UJ

1A-253

000247

WHC-SD-WM-DP-053  
ADDENDUM 1/REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analysis must be performed with every analytical batch or every twenty samples, whichever is more frequent. This requirement may be satisfied with the analysis of an MS/MSD sample. Method or program DQO specified control limits shall be applied to sample results where they exist, otherwise the Relative Percent Difference (RPD) shall be less than 20% for water samples (<35% for soils) if the sample result is greater than 5X times the RDL. If the sample result is less than 5 times the RDL, the difference between the primary and duplicate results must be less than the RDL for water samples and less than 2 times the RDL for soils. If both sample and duplicate results are below the Method Detection Limit (MDL) or Sample Quantitation Limit (SQL), then no control limit applies.

**ACTION:** All QC criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-254

248  
00025001/1/94

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: MATRIX SPIKES/TRACERS/CARRIERS

**COMMENTS:** Matrix spikes, tracers, and carriers are used in radiochemical analyses to indicate overall accuracy for a given matrix. Matrix spikes are not required for GEA or Total (Gross) Beta analyses. The control limits for matrix spikes, carriers, or tracers is less than three standard deviations of normal operating conditions. Results outside these limits are qualified as estimated or unusable based on the judgement of the reviewer.

**ACTION:** All carrier recoveries were acceptable.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-255

000249

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: PREPARATION BLANKS

**COMMENTS:** Preparation blanks were evaluated for the presence of contaminants. At least one preparation blank is required for each sample batch. If the concentration of analytes in the blanks exceeded 20% of the sample concentration, the associated sample results were qualified as estimated. If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated. In this case, the blank result was less than the MDA.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-256

250  
000248  
1/13/93

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: LABORATORY CONTROL SAMPLES

**COMMENTS:** The Laboratory Control Sample (LCS) is a monitor of the overall performance of analytical method, including sample preparation. An LCS must be analyzed with each batch. The LCS recoveries must be within 3 times the standard deviation of normal operating conditions. Results outside these limits are qualified as estimated or unusable depending on the judgement of the validator.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
-----------------	--------------------	------------------------

1A-257

000251

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D. J. Smith

Date 1/7/94

QC Check: OTHER QUALITY CONTROL

**COMMENTS:** The radiochemical data was examined for compliance with specific project Data Quality Objectives and the Statement of Work. Any trends observed in the performance of an instrument, method, or laboratory of the course of the analysis are noted.

**ACTION:** All analyses were performed according to the requirements of the QAPP and TPP. No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-258

000252

RADIOCHEMICAL DATA ASSESSMENT

DATE	<u>1-4-94</u>	SAMPLES/MATRIX	<u>V34/LIQUID</u>
REVIEWED BY	<u>D.J. SMITH</u>		
LABORATORY	<u>222-S LABORATORY</u>		
CASE #	<u>242 EVAPORATOR</u>		
SDG #	<u>TANK 241-AP-107</u>		

*ES*  
*1/14/94*

DATA ASSESSMENT SUMMARY

	TOTAL <u>Np<sup>237</sup></u>	<u>Pu<sup>239/240</sup></u>	<u>Am<sup>241</sup></u>	<u>Pu<sup>238</sup></u>
1. <u>Chain of Custody</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
2. <u>Initial Calibration</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
3. <u>Efficiency Checks</u>	<u>X</u>	<u>X</u>	<u>X</u>	<u>X</u>
4. <u>Background Checks</u>	<u>X</u>	<u>X</u>	<u>X</u>	<u>X</u>
5. <u>Preparation Blanks</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
6. <u>MS/Tracers/Carriers</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
7. <u>Duplicate Analysis</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>
8. <u>LCS</u>	<u>0</u>	<u>0</u>	<u>0</u>	<u>0</u>

0 = data had no problems  
X = minor problems, data may be qualified  
M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: All results were qualified as estimated (UJ) for missing efficiency and background check results and control charts.

NOTES: \_\_\_\_\_

o Refer to the corresponding attachments for explanation of any problems.

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL DATA ASSESSMENT

DATE January 7, 1993 SAMPLES/MATRIX V-34/Liquid  
 REVIEWED BY D. J. Smith *DJS 1/13/93*  
 LABORATORY 222-S  
 CASE # 241-AP-107  
 SDG # 241-AP-107

DATA ASSESSMENT SUMMARY

	<u>Cm243/244</u>			
1. <u>Chain of Custody</u>	<u>0</u>			
2. <u>Requested/Reported Anal.</u>	<u>0</u>			
3. <u>Holding Times</u>	<u>0</u>			
4. <u>Calibration</u>	<u>0</u>			
5. <u>Efficiency Checks</u>	<u>X</u>			
6. <u>Background Checks</u>	<u>X</u>			
7. <u>Duplicate Analysis</u>	<u>0</u>			
8. <u>MS/Tracers/Carriers</u>	<u>0</u>			
9. <u>Analytical Blanks</u>	<u>0</u>			
10. <u>LCS</u>	<u>0</u>			
11. <u>Other QC Checks</u>	<u>0</u>			

0 = data had no problems  
 X = minor problems, data may be qualified  
 M = data qualified due to major problems/some data may be unusable

OVERALL ASSESSMENT: The overall quality of the data is good. All results were qualified as estimated for missing efficiency and background check documentation.

NOTES: \_\_\_\_\_

o Refer to the corresponding attachments for explanation of any problems.

1A-260

253.1

RADIOCHEMICAL QC

Name D.J. SMITH

Date 1-4-94

QC Check: CHAIN OF CUSTODY

**COMMENTS:** The sample was collected by WHC on 08-1-93 and transferred in chilled containers without incident to the 222-S Laboratory for the following analyses: Np<sup>237</sup>, Pu<sup>239/240</sup>, Am<sup>241</sup>. The 222-S Laboratory received the samples on 08-02-93 and analysis took place on 09-02-93 and 09-10-93, within holding time specifications.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-261

000254

RADIOCHEMICAL QC

Name D.J. SMITH

Date 1-4-94

QC Check: INITIAL CALIBRATION

**COMMENTS:** The data packages are reviewed to verify that the instrument was calibrated within the time period specified by the laboratory standard operating procedure or manufacturer's instruction. Instrument efficiencies are determined from the initial calibration. If the instrument was not calibrated within the specified time period, all associated results are qualified as unusable.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-262

000255

RADIOCHEMICAL QC

Name D.J. SMITH

Date 01-04-94

QC Check: EFFICIENCY CHECKS

COMMENTS: Efficiency checks are counted to ensure that acceptable instrument performance is maintained on a day to day basis. Efficiency check data is reviewed to verify that the frequency requirements were met and that all results were within control limits (< 3 sigma). If efficiency QC criteria are not met, sample results are qualified as estimated. Efficiency checks were not provided by the laboratory.

ACTION: Qualify all results as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V34	Np <sup>237</sup>	<1.48 E-5/UJ
V34	Pu <sup>239/240</sup>	<5.34 E-4/UJ
V34	Am <sup>241</sup>	<6.37 E-4/UJ
V34	Pu <sup>238</sup>	<5.97 E-4/UJ
V34	Cm <sup>243/244</sup>	<6.37 E-4/UJ

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D.J. SMITH

Date 01-04-94

QC Check: BACKGROUND CHECKS

**COMMENTS:** Background radiation is measured by counting a simulated sample or source which identical to the actual sample except for the absence of radioactivity from a sample source. Background checks must be acquired for each detector system on a regular basis. The frequency of background checks is dependent on the sample count time.

<u>Count Time</u>	<u>Background Frequency</u>
0-1 hour	1 per 8 hours
1-8 hours	1 per 24 hours
>8 hours	1 per week

Background checks should not deviate more than 3 times the standard deviation of normal operating conditions. If the background results are outside of the specified frequency or control limits, sample results are qualified as estimated. Background check results were not provided by the laboratory.

**ACTION:** Qualify all results as estimated.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
V34	Np <sup>237</sup>	<1.48 E-5/UJ
V34	Pu <sup>239/240</sup>	<5.34 E-4/UJ
V34	Am <sup>241</sup>	<6.37 E-4/UJ
V34	Pu <sup>238</sup>	<5.97 E-4/UJ
V34	Cm <sup>243/244</sup>	<6.37 E-4/UJ

1A 264

000257

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D.J. SMITH

Date 01-04-94

QC Check: PREPARATION BLANKS

**COMMENTS:** Preparation blanks were evaluated for the presence of contaminants. At least one preparation blank is required for each sample batch. If the concentration of analytes in the blanks exceeded 5 times the sample concentration, the associated sample results were qualified as nondetected (U). If the absolute value of any negative blank values exceeded the Instrument Detection Limit (IDL), non-detects were qualified as estimated (UJ) and positive results within 2 times the absolute value of the blank value as estimated.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-265

000258

WHC-SD-WM-DP-053  
ADDENDUM 1A REV. 0

RADIOCHEMICAL QC

Name D.J. SMITH

Date 01-04-94

QC Check: MATRIX SPIKES/TRACERS/CARRIERS

**COMMENTS:** Matrix spikes, tracers, and carriers are used in radiochemical analyses to indicate overall accuracy for a given matrix. Matrix spikes are not required for GEA or Total (Gross) Beta analyses. The control limit for matrix spikes, carriers, or tracers is less than three standard deviations of normal operating conditions. Results outside these limits are qualified as estimated or unusable based on the judgement of the reviewer.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-266

000259

RADIOCHEMICAL QC

Name D.J. SMITH

Date 01-04-94

QC Check: DUPLICATE ANALYSIS

**COMMENTS:** Duplicate analyses are performed to monitor the precision of the method. Duplicate results should be within 3 sigma of normal operating conditions. If either the sample or duplicate is below the Minimum Detectable Activity (MDA) then no control limit applies. All results outside the control limit are qualified as estimated.

**ACTION:** All QC criteria were met.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-267

000250

RADIOCHEMICAL QC

Name D.J. SMITH

Date 01-04-94

QC Check: LABORATORY CONTROL SAMPLES

**COMMENTS:** The Laboratory Control Sample (LCS) is a monitor of the overall performance of analytical method, including sample preparation. An LCS must be analyzed with each batch. The LCS recoveries must be within 3 times the standard deviation of normal operating conditions. Results outside these limits are qualified as estimated or unusable depending on the judgement of the validator.

**ACTION:** No action is required.

<u>sample #</u>	<u>constituent</u>	<u>value/qualifier</u>
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1A-268

000061

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CHAINS OF CUSTODY

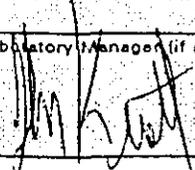
1A-270

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REQUEST FOR SPECIAL ANALYSIS (RSA)

RSA No.

1626

1. Sample Origin <b>107-AP</b>		2. Date Submitted <b>8/2/93</b>		4. Requester Name <b>George L Miller</b>		6. Charge Code/Work Package <b>N1417</b>	
		3. Submitted By <b>George L Miller</b>		5. Requester Phone/MSIN <b>373-4739 / TG-06</b>		7. Date Required <b>9/3/93</b>	
8. Customer ID <b>107-AP-COMP</b>	Collected		9. Laboratory ID <b>V34</b>		12. Number of Samples <b>1</b>	13. Volume of Samples <b>100ml</b>	14. Protocols <input type="checkbox"/> None <input checked="" type="checkbox"/> RCRA <input type="checkbox"/> NQA-1 <input type="checkbox"/> Other <input type="checkbox"/> CERCLA
	Date	Time					
	<b>N/A</b>						<b>Techno</b>
				15. Sample Type <input type="checkbox"/> Solid <input type="checkbox"/> Gas <input type="checkbox"/> Solution <input type="checkbox"/> Other (specify) _____ <input type="checkbox"/> Soil <input checked="" type="checkbox"/> Water <input type="checkbox"/> Sludge <input type="checkbox"/> Slurry <input type="checkbox"/> Waste <input type="checkbox"/> Oil			
				16. Storage Requirements <input checked="" type="checkbox"/> None <input type="checkbox"/> Specify _____			
				17. Process Knowledge/Known Listed Wastes <input type="checkbox"/> Unknown <input checked="" type="checkbox"/> Known (Please attach list) <b>Evaporator Feed</b> <input checked="" type="checkbox"/> Previously submitted for this Project ( <b>Similar to 107-AP</b> )			
10. Determination		11. Expected Range		18. Survey <span style="float:right">Dose/Rat</span>			
<b>Appearance</b>				HPT Signature _____			
<b>C-14</b>							
<b>I-129</b>				19. Disposition of Waste <input type="checkbox"/> Return to Client/Location _____ <input type="checkbox"/> Dispose per 222-S Procedures <input checked="" type="checkbox"/> Other per TPT: <b>WHC-SD-WM-TPP-048</b>			
<b>H-3</b>				20. Additional Information Sample 107-AP-COMP is to be generated as a composite (equal volumes) from samples 107-AP-A, 107-AP-B, 107-AP-C, 107-AP-D, and 107-AP-E  This sample requires Sample, Sample Duplicate, Sample Spike and Spike Duplicate for C-14, I-129, H-3, Pu-239/240, Cm-244, Am-241, Np-237, AT, TB, Sr-90, Tc-99			
<b>Acid Digest</b>							
<b>-GEA</b>							
<b>-Pu-239/240</b>							
<b>-Am-241</b>							
<b>-Cm-244</b>							
<b>-Np-237</b>							
<b>-TA</b>							
<b>-TB</b>				22. Assigned Custodial Group (select one) <input type="checkbox"/> PCL <input checked="" type="checkbox"/> PDSU <input type="checkbox"/> ENV <input type="checkbox"/> Process			
<b>-Sr-90</b>				24. Laboratory Manager (if required) 			
<b>-Tc-99</b>							
21. Chain of Custody <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes Number: <b>Internal 122</b>				23. Sample Received By: <b>Spencer Cook</b> <b>1A-272</b>			
Date: <b>08-10-93</b>		Time: <b>1115</b>		000268		12/15/93	

REQUEST FOR SPECIAL ANALYSIS (RSA)

RSA No.

1625

1. Sample Origin <b>107-AP</b>		2. Date Submitted <b>8/2/93</b>		4. Requester Name <b>George L Miller</b>		6. Charge Code/Work Package <b>NIL17</b>	
		3. Submitted By <b>George L Miller</b>		5. Requester Phone/MSIN <b>373-4739 /TG-06</b>		7. Date Required <b>9/3/93</b>	
8. Customer ID <b>107-AP-FB</b>	Collected		9. Laboratory ID <b>V31</b>	12. Number of Samples <b>1</b>	13. Volume of Samples <b>Approx 100 ml</b>	14. Protocols <i>Technology</i>	
	Date <b>8/1/93</b>	Time <b>1005</b>				<input type="checkbox"/> None <input checked="" type="checkbox"/> RCRA	<input type="checkbox"/> Other
				15. Sample Type			
				<input type="checkbox"/> Solid <input type="checkbox"/> Soil <input type="checkbox"/> Slurry			
				<input type="checkbox"/> Gas <input checked="" type="checkbox"/> Water <input type="checkbox"/> Waste			
				<input type="checkbox"/> Solution <input type="checkbox"/> Sludge <input type="checkbox"/> Oil			
				<input type="checkbox"/> Other (specify) _____			
				16. Storage Requirements			
				<input checked="" type="checkbox"/> None			
				<input type="checkbox"/> Specify _____			
				17. Process Knowledge/Known Listed Wastes			
				<input type="checkbox"/> Unknown			
				<input checked="" type="checkbox"/> Known (Please attach list) <b>Water (from Lab)</b>			
				<input checked="" type="checkbox"/> Previously submitted for this Project ( <b>same as 101-AP-FB</b> )			
10. Determination		11. Expected Range		18. Survey			
<b>Uranium - Fluorimetric</b>				Dose Rate			
<b>OH<sup>-</sup></b>				HPT Signature			
<b>Hg</b>				19. Disposition of Waste			
<b>Acid Digest</b>				<input type="checkbox"/> Return to Client/Location _____			
<b>-ICP</b>				<input type="checkbox"/> Dispose per 222-S Procedures			
<b>-As</b>				<input checked="" type="checkbox"/> Other per TPP: <b>WHC-SD-WM-TPP-048</b>			
<b>-Se</b>							
<b>-GEA</b>							
<b>-TA</b>							
<b>-TB</b>							
<b>Water Digest</b>				20. Additional Information			
<b>-IC</b>				Sample requires: { Sample Duplicate			
<b>-NH<sub>4</sub></b>				Shipping # <b>R3630</b> is Sample ID# <b>107-AP-FB</b>			
<b>-CN</b>							
<b>-TIC</b>							
21. Chain of Custody				22. Assigned Custodial Group (select one)			
<input type="checkbox"/> No				<input type="checkbox"/> PCL <input checked="" type="checkbox"/> TDSU <input type="checkbox"/> ENV <input type="checkbox"/> Process			
<input checked="" type="checkbox"/> Yes Number: <b>(No Custody Seal # on CufC)</b>				<b>123</b>			
23. Sample Received By: <b>Eric P. ...</b>				24. Laboratory Manager (if required)			
Date: <b>8/2/93</b> Time: <b>1730</b>				<b>000264</b> Date: <b>10/15/93</b>			

REQUEST FOR SPECIAL ANALYSIS (RSA)

RSA No.

1624

Sample Origin <b>107-AP</b>	2. Date Submitted <b>8/2/93</b>	4. Requester Name <b>George L Miller</b>	6. Charge Code/Work Package <b>NIL17</b>
	3. Submitted By <b>George L Miller</b>	5. Requester Phone/MSN <b>373-4739 / T6-06</b>	7. Date Required <b>9/3/93</b>

B. Customer ID	Collected		9. Laboratory ID	12. Number of Samples <b>3</b>	13. Volume of Sample <b>Approx. 100 ml</b>	14. Protocols <input type="checkbox"/> None <input checked="" type="checkbox"/> RCRA Technology <input type="checkbox"/> NOA-1 <input type="checkbox"/> Other <input type="checkbox"/> CERCLA
	Date	Time				

<b>107-AP-C</b>	<b>8/1/93</b>	<b>1209 PM</b>	<b>V 26</b>	15. Sample Type <input type="checkbox"/> Solid <input type="checkbox"/> Soil <input type="checkbox"/> Slurry <input type="checkbox"/> Gas <input checked="" type="checkbox"/> Water <input type="checkbox"/> Waste <input type="checkbox"/> Solution <input type="checkbox"/> Sludge <input type="checkbox"/> Oil <input type="checkbox"/> Other (specify) _____
<b>107-AP-D</b>	<b>8/1/93</b>	<b>1220 PM</b>	<b>V 27</b>	
<b>107-AP-E</b>	<b>8/1/93</b>	<b>1145 AM</b>	<b>V 28</b>	

10. Determination	11. Expected Range	16. Storage Requirements <input checked="" type="checkbox"/> None <input type="checkbox"/> Specify _____
		17. Process Knowledge/Known Listed Wastes <input type="checkbox"/> Unknown <input checked="" type="checkbox"/> Known (Please attach list) <b>Evaporator Feed</b> <input checked="" type="checkbox"/> Previously submitted for this Project <b>Similar to 101-1</b>
		18. Survey _____ Date Rate _____ HPT Signature _____
		19. Disposition of Waste <input type="checkbox"/> Return to Client/Location _____ <input type="checkbox"/> Dispose per 222-S Procedures <input checked="" type="checkbox"/> Other <b>per TPP: WHC-SD-WM-TPP-048</b>

20. Additional Information <b>top 12/93</b> Sample A requires: { Sample Duplicate Sample D requires: { Sample Duplicate Sample E requires: { Sample Duplicate Shipping # R3623 is Sample ID # 107-AP-C Shipping # R3625 is Sample ID # 107-AP-D Shipping # R3627 is Sample ID # <sup>from 8/1/93</sup> 107-AP-E
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22. Assigned Custodial Group (select one) <input type="checkbox"/> PCL <input checked="" type="checkbox"/> PSDU <input type="checkbox"/> ENV <input type="checkbox"/> Process <b>124</b>
---

Chain of Custody <input type="checkbox"/> No <input checked="" type="checkbox"/> Yes Number: <b>Seal # 3353 (107-AP-C)</b> <b>Seal # 3362 (107-AP-D)</b> <b>Seal # 3357 (107-AP-E)</b>
--

23. Sample Received By: <b>John Pulick 1A-274</b> Date: <b>8/2/93</b> Time: <b>1730</b>	24. Laboratory Manager (if required) <b>John Pulick</b> <b>000265</b> Date: <b>12/15/93</b>
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REQUEST FOR SPECIAL ANALYSIS (RSA)

RSA No.

1623

Sample Origin <b>107-AP</b>		2. Date Submitted <b>8/2/93</b>		4. Requester Name <b>George L Miller</b>		6. Charge Code/Work Package <b>N1417</b>	
		3. Submitted By <b>George L Miller</b>		5. Requester Phone/MSIN <b>373-4739 / TG-06</b>		7. Date Required <b>9/3/93</b>	
B. Customer ID	Collected		9. Laboratory ID	12. Number of Samples <b>2</b>	13. Volume of Samples <b>Approx. 100 ml</b>	14. Protocols	
	Date	Time				<input type="checkbox"/> None	<input checked="" type="checkbox"/> RCRA <i>Technogy</i>
<b>107-AP-A</b>	<b>8/1/93</b>	<b>1040 AM</b>	<b>V21</b>			<input type="checkbox"/> CERCLA	
<b>107-AP-B</b>	<b>8/1/93</b>	<b>1024 AM</b>	<b>V23</b>				
				15. Sample Type			
				<input type="checkbox"/> Solid <input type="checkbox"/> Soil <input type="checkbox"/> Slurry <input type="checkbox"/> Gas <input checked="" type="checkbox"/> Water <input type="checkbox"/> Waste <input type="checkbox"/> Solution <input type="checkbox"/> Sludge <input type="checkbox"/> Oil <input type="checkbox"/> Other (specify) _____			
				16. Storage Requirements			
				<input checked="" type="checkbox"/> None <input type="checkbox"/> Specify _____			
				17. Process Knowledge/Known Listed Wastes			
				<input type="checkbox"/> Unknown <input checked="" type="checkbox"/> Known (Please attach list) <b>Evaporator feed</b> <input checked="" type="checkbox"/> Previously submitted for this Project ( <b>similar to 101-AP</b> )			
10. Determination		11. Expected Range		18. Survey <span style="float:right">Dose Rate</span>			
<b>Appearance</b>				HPT Signature _____			
<b>DSC</b>							
<b>Sp Gravity</b>				19. Disposition of Waste			
<b>Uranium-Fluorometric</b>				<input type="checkbox"/> Return to Client/Location _____ <input type="checkbox"/> Dispose per 222-S Procedures <input checked="" type="checkbox"/> Other <b>per TPP: WHC-SD-WH-TPP-048</b>			
<b>OH<sup>-</sup></b>				20. Additional Information			
<b>Hg</b>				Sample A requires: { Sample Duplicate, Spike, Spike Duplicate Sample B requires: { Sample Duplicate			
<b>Acid Digest</b>				Shipping # R3619 is Sample ID # 107-AP-A			
<b>- ICP</b>				Shipping # R3621 is Sample ID # 107-AP-B			
<b>- As</b>							
<b>- Se</b>							
<b>Water Digest</b>							
<b>- IC</b>							
<b>- NH<sub>4</sub></b>							
<b>- CN</b>							
<b>- TIC</b>							
Chain of Custody:				22. Assigned Custodial Group (select one)			
<input type="checkbox"/> No <input checked="" type="checkbox"/> Yes    Number: <b>No Seal # for Shipping # R3619</b> <b>Seal # 3363 (107-AP75)</b>				<input type="checkbox"/> PCL <input checked="" type="checkbox"/> PSDU <input type="checkbox"/> ENV <input type="checkbox"/> Process <b>125</b>			
23. Sample Received By: <b>JR Pilechik 1A-275</b>				24. Laboratory Manager (if required)			
Date: <b>08-02-93</b> Time: <b>1730</b>				<b>J. K. Kuest</b> <b>000266</b> Date: <b>12/15/93</b>			

REQUEST FOR SPECIAL ANALYSIS (RSA)

RSA No.

11627

Sample Origin <b>107-AP</b>	2. Date Submitted <b>8/2/93</b>	4. Requester Name <b>George L Miller</b>	6. Charge Code/Work Package <b>8/2/93 Jan NIP17</b>
	3. Submitted By <b>George L Miller</b>	5. Requester Phone/MSIN <b>373-4739 /TG-06</b>	7. Date Required <b>8/15/93</b>

8. Customer ID <b>VOA</b>	Collected		9. Laboratory ID <b>V35</b>	12. Number of Samples <b>1</b>	13. Volume of Samples <b>100ml</b>	14. Protocols	
	Date <b>8/1/93</b>	Time <b>1115</b>				<input type="checkbox"/> None	<input checked="" type="checkbox"/> RCRA

15. Sample Type

<input type="checkbox"/> Solid	<input type="checkbox"/> Soil	<input type="checkbox"/> Slurry
<input type="checkbox"/> Gas	<input checked="" type="checkbox"/> Water	<input type="checkbox"/> Waste
<input type="checkbox"/> Solution	<input type="checkbox"/> Sludge	<input type="checkbox"/> Oil
<input type="checkbox"/> Other (specify) _____		

16. Storage Requirements

None

Specify Refrigerated to 4°C in VOA cabinet

17. Process Knowledge/Known Listed Wastes

Unknown

Known (Please attach list) Evaporator Feed

Previously submitted for this Project

10. Determination <b>Volatile Organic Analysis</b>	11. Expected Range	18. Survey	Dose rate
		_____ HPT Signature	

19. Disposition of Waste

Return to Client/Location \_\_\_\_\_

Dispose per 222-S Procedures

Other \_\_\_\_\_

20. Additional Information

**Run Sample in Duplicate for VOA by both CLP and RCRA methods.**

**Two week sample holding time.**

**Provide analytical data to HASM in a timely manner**

**Shipping # R3510 is Sample ID # VOA**

Chain of Custody

No

Yes Number: **Seal # 3357 126**

22. Assigned Custodial Group (select one)

PCL  PDSU  ENV  Process X Line

23. Sample Received By: **GR Puluech**

Date: **8/2/93** Time: **17:30**

24. Laboratory Manager (if required)

**John Kust** **000267** Date: **12/15/93**

WHC-SD-WM-DP-053 REV0 ADDENDUM 1A

COPY  
COPY

CHAIN OF CUSTODY			
Company Contact	RL WRIGHT	Telephone	373-3552
Bill of Lading No.	NA	Offsite Property No.	NA
Method of Shipment	B-PIAST Sample TRUCK		
Shipped to	277-S Lab		

SAMPLING INFORMATION			
Sample Collected by	CJL/MCKRT	Date	8-1-93
Sample Locations	107-AP 1(SW)	Custody Seal #	3354
Remarks	NONE		
Ice Chest or Sample Pkg No.	ICE CREAM CARTON	Field Logbook and Page No.	N/A

SUPERVISION REVIEW: R. Wright DATE: 8-1-93

SAMPLE IDENTIFICATION

Sample Number	Sample Schedule Number
<u>R 3627/V-28</u>	<u>NA</u>

CHAIN OF POSSESSION

Relinquished by: <u>R. Wright</u>	Received by: <u>A. [Signature]</u>	Date/Time: <u>8/1/93 1700</u>
Relinquished by: <u>A. [Signature]</u>	Received by: <u>R. [Signature]</u>	Date/Time: <u>8/2/93 1735</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

**COPY**

CHAIN OF CUSTODY			
Company Contact	RL WRIGHT	Telephone	373-3552
Bill of Lading No.	NA	Offsite Property No.	NA
Method of Shipment	B-PLANT SAMPLE TRUCK		
Shipped to	222-S LAB		
SAMPLING INFORMATION			
Sample Collected by	Caylor/MERT	Date	8-1-93
Sample Locations	107-AD	Custody Seal #	
Remarks	NONE		
Ice Chest or Sample Bag No.	ICE CREAM CARTON	Field Logbook and Page No.	N/A

SUPERVISION REVIEW: RJ Wright DATE: 8/2/93

SAMPLE IDENTIFICATION	
Sample Number	Sample Schedule Number
<u>R3630/V-31</u>	<u>NA</u>

CHAIN OF POSSESSION		
Relinquished by: <u>RJ Wright</u>	Received by: <u>A. Hermulen</u>	Date/Time: <u>8/2/93 1200</u>
Relinquished by: <u>A. Hermulen</u>	Received by: <u>C. P. Black</u>	Date/Time: <u>8/2/93 1730</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

1A-278

# TANK FARM PLANT OPERATING PROCEDURE

WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

COPY

CHAIN OF CUSTODY			
Company Contact:	RL WRIGHT	Telephone:	323-3552
Bill of Lading No.:	NA	Offsite Property No.:	NA
Method of Shipment:	B-PLANT SAMPLE TRUCK		
Shipped to:	222-S LAB		
SAMPLING INFORMATION			
Sample Collected by:	Caylor/MCKT	Date:	8-1-93
Sample Locations:	107-AP/1 (E)	Custody Seal #:	3362
Remarks:	NONE		
Ice Chest or Sample Pkg No.:	ICE CREAM CARTON	Field Logbook and Page No.:	N/A

SUPERVISION REVIEW: *RJ Wright* DATE: 8-2-93

SAMPLE IDENTIFICATION	
Sample Number	Sample Schedule Number
<u>R3625/V-27</u>	<u>NA</u>

CHAIN OF POSSESSION		
Relinquished by: <i>RJ Wright</i>	Received by: <i>M. Hummel</i>	Date/Time: <i>8/2/93 1720</i>
Relinquished by: <i>M. Hummel</i>	Received by: <i>CR P. Lusk</i>	Date/Time: <i>8/2/93 1720</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

/A-279

**COPY**

CHAIN OF CUSTODY				ADDENDUM 1A
Company Contact:	RL WRIGHT	Telephone:	713-3552	
Bill of Lading No.:	NA	Offsite Property No.:	NA	
Method of Shipment:	B-Plant Sample TRUCK			
Shipped to:	222-S LAG (WHC)			

SAMPLING INFORMATION					
Sample Collected by:	Caylor/MART	Date:	8-1-93	Time:	11:40 AM
Sample Locations:	107-AP (NW)	Custody Seal #:			
Remarks:	LOW				
Ice Chest or Sample Pig No.:	ICE CREAM CARTON	Field Logbook and Page No.:	N/A		

SUPERVISION REVIEW: Rf Wright DATE: 8-1-93

SAMPLE IDENTIFICATION	
Sample Number	Sample Schedule Number
<u>R3619/V-21</u>	<u>NA</u>

CHAIN OF POSSESSION		
Relinquished by: <u>Rf Wright</u>	Received by: <u>A. Verrill</u>	Date/Time: <u>8/2/93 1700</u>
Relinquished by: <u>A. Verrill</u>	Received by: <u>Ch. P. ...</u>	Date/Time: <u>8/2/93 1730</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

1A-280

# TANK FARM PLANT OPERATI. G PROCEDURE

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

COPY

CHAIN OF CUSTODY			
Company Contact	RL WRIGHT	Telephone	373-3552
Bill of Lading No.	NA	Offsite Property No.	NA
Method of Shipment	B-PLANT SAMPLE TRUCK		
Shipped to	22-S (WHC)		
SAMPLING INFORMATION			
Sample Collected by	CAYLOR/MARKT	Date	8-1-93
Sample Locations	107-A1 (E)	Custody Seal #	3353
Remarks	NONE		
Ice Chest or Sample Pkg No.	ICE CREAM CARTON	Field Logbook and Page No.	N/A

SUPERVISION REVIEW: RL Wright

DATE: 8-1-93

### SAMPLE IDENTIFICATION

Sample Number	Sample Schedule Number
<u>R3623/V-26</u>	<u>NA</u>

### CHAIN OF POSSESSION

Relinquished by:	Received by:	Date/Time:
<u>RL Wright</u>	<u>St. Hummel</u>	<u>8/2/93 1700</u>
Relinquished by:	Received by:	Date/Time:
<u>St. Hummel</u>	<u>A.R. Paluck</u>	<u>8/2/93 1735</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

1A-281

126.5

000272

# TANK FARM PLANT OPERATING PROCEDURE

WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

**COPY**

CHAIN OF CUSTODY			
Company Contact	RL WRIGHT	Telephone	373-3552
Bill of Lading No.	NA	Offsite Property No.	NA
Method of Shipment	B-PLANT SAMPLE TRUCK		
Shipped to	222-S LAB		
SAMPLING INFORMATION			
Sample Collected by	Coyler/McKee	Date	8-1-93
Sample Locations	107-AP/1 (NW)	Custody Seal #	336.3
Remarks	NONE		
Ice Chest or Sample Pkg No.	ICC CREAM CARTON	Field Logbook and Page No.	N/A

SUPERVISION REVIEW: RJ Wright DATE: 8-2-93

SAMPLE IDENTIFICATION	
Sample Number	Sample Schedule Number
<u>R3621/V-23</u>	<u>NA</u>

CHAIN OF POSSESSION		
Relinquished by: <u>RJ Wright</u>	Received by: <u>A. Mendenhall</u>	Date/Time: <u>8/2/93 1700</u>
Relinquished by: <u>A. Mendenhall</u>	Received by: <u>C. P. Plunk</u>	Date/Time: <u>8/2/93 1730</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

1A-282

# TANK FARM PLANT OPERATING PROCEDURE

WHC-SD-WM-DP-053 REV 0

COPY

CHAIN OF CUSTODY				ADDENDUM 1A	
Company Contact	RL WRIGHT	Telephone	373-3552		
Bill of Lading No.	NA	Offsite Property No.	NA		
Method of Shipment	B-Plant Sample TRUCK				
Shipped to	222-S LAB				
SAMPLING INFORMATION					
Sample Collected by	CAYLOR/MICHAEL	Date	8-1-93	Time	1115
Sample Locations	107-2A 1(SW)	Custody Seal #	3357		
Remarks	NONE				
Ice Chest or Sample Pkg No.	ICE CREAM CARTON	Field Logbook and Page No.	N/A		

SUPERVISION REVIEW: *R. Wright* DATE: 8-2-93

### SAMPLE IDENTIFICATION

Sample Number	Sample Schedule Number
<u>R3910/V-35</u>	<u>NA</u>

### CHAIN OF POSSESSION

Relinquished by: <i>R. Wright</i>	Received by: <i>S. Munnich</i>	Date/Time: <i>8/2/93 1700</i>
Relinquished by: <i>S. Munnich</i>	Received by: <i>C. R. Dulich</i>	Date/Time: <i>8/2/93 1730</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

1A-283

Document No.	Rev/Mod.	Page
TO-080-030	C-4	21

126.7

000274

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SUPPLEMENTAL INFORMATION

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Ø5] From: Scot L Fitzgerald 11/5/93 12:27PM (1917 bytes: 29 ln)  
To: George L Miller  
Subject: V34 Total alpa spike recoveries WHC-SD-WM-DP-053 REV 0 ADDENDUM 1A

----- Message Contents -----  
T SAY IT --- Write It! DATE: November 5, 1993

---

TO: George Miller T6-06  
FROM: Scot Fitzgerald T6-50 Telephone: 373-2378  
cc:  
SUBJECT: Total alpha spike recovery for sample V-34

---

As you know sample V-34 has been run a total of three times at present. In the initial run the spike and spike duplicate recoveries were 68.7% and 64.3% respectively. These values did not meet the 75-125% recovery criteria so the sample was sent out for rerun. The spike and spike duplicate recoveries for the rerun were 42.5% and 87.9% respectively. The large variance between the two recoveries indicated a probable tech error so the sample was sent out for a second rerun. The spike and spike duplicate recoveries for the second rerun were 64.0% and 75.3% respectively. As you can see, one of these values again falls outside the recovery criteria. During this rerun however, the tech noted that there were solids present on the counting planchet after sample evaporation. After discussing the sample appearance with the tech involved, I have concluded that the low spike recoveries for the initial run and the second rerun are most likely due to self adsorption. Since the sample values returned for all three runs are below are detection limits, a third rerun of this sample does not seem necessary  
MACPATH-GEF1000

1A-287

128

000276

Narrative for NH<sub>4</sub><sup>+</sup> Analysis for Tank 107-AP

This narrative concerns Batch 1503 of tank 107-AP NH<sub>4</sub><sup>+</sup> analysis. The detection limit of the blank is not the same as the detection limits of the samples analyzed in this batch. The spike and spike duplicate are at highest acceptable recovery as set in the TTP. Due to expired holding time, the batch will not be re-analyzed.

The detection limit is inversely proportional to volume of sample used in the analysis. This was the first batch of 107-AP samples run for NH<sub>4</sub><sup>+</sup>. Assuming that this tank contained similar concentrations of NH<sub>4</sub><sup>+</sup> as the previous evaporator tank (101-AP) analyzed, approx. 2000 ppm, the analyst began with an appropriate sample size (0.500ml). This volume was first used for the blank, then the first analysis of the first sample. After the first analysis, the analyst made an adjustment to a larger sample size (1.000ml) for the duplicate analysis of the first sample, and the rest of the batch, to improve the detection limit. The analyst was instructed not to exceed a 1.000ml sample size to conserve the limited amount of sample available. There was still no analyte detected, so the results were reported.

This batch should be accepted as valid data. The same water and the same amount of reagent was used for the samples as for the blank, and the samples were below detection limit for NH<sub>4</sub><sup>+</sup> using the 1.000ml volume. This shows that the blank was not contaminated at the lower detection limit. The 125% recovery translates to a 219ppm bias high. The blank contributed <40ppm. Again, if spike recovery was high and the samples were still below detection, there is no reason to suspect the data is not valid.

Robert W. Schroeder  
cognizant scientist

*Robert W. Schroeder*

11-1-93

1A-288

129

000277

[21] From: John F Orourke at ~WHC338 11/19/93 3:07PM (813 bytes: 13 ln)  
To: George L Miller at ~WHC168  
cc: John G Kristofzski at ~WHC168, Richard J (Dick) Nicklas, Brian H Von Bargaen,  
John F Orourke  
Subject: 107-AP DATA PACKAGE

----- Message Contents -----

George,

WHC-SD-WM-DP-053 REV 0

Our group is not currently preparing documentation for the second evaporator campaign. Therefore, the LCCS printouts specified in the Statement of Work for tank 107-AP are not presently required. I'll let you know if this situation changes.

ADDENDUM 1A

I look forward to seeing the preliminary data package for 107-AP in the mid-December time frame.

John O'Rourke  
373-2977

1A 289'

130

000278

DSC STD  
11.031 mg

Rate: 10.0 °C/min

File: 00069.001

Ident:

DSC METTLER  
222-S Laboratory

20-Aug-93  
RW 8-31-93

exo >

50.0 mW

157

DSC STD

Integration

Delta H 2276 mJ

206.4 J/g

Peak 250.7 °C

6.6 mW

V-19

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

SIGNATURE ABOVE REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT  
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 157 TO 162

Robby Wendi (signature)

14290

000279

100.

200.

300.

400.

°C

V21  
10.075 mg

Rate: 10.0 °C/min

File: 00073.001 DSC METTLER 31-Aug-93  
Ident: 222-S Laboratory

<exo>

V21

50. mW

158

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

100.

200.

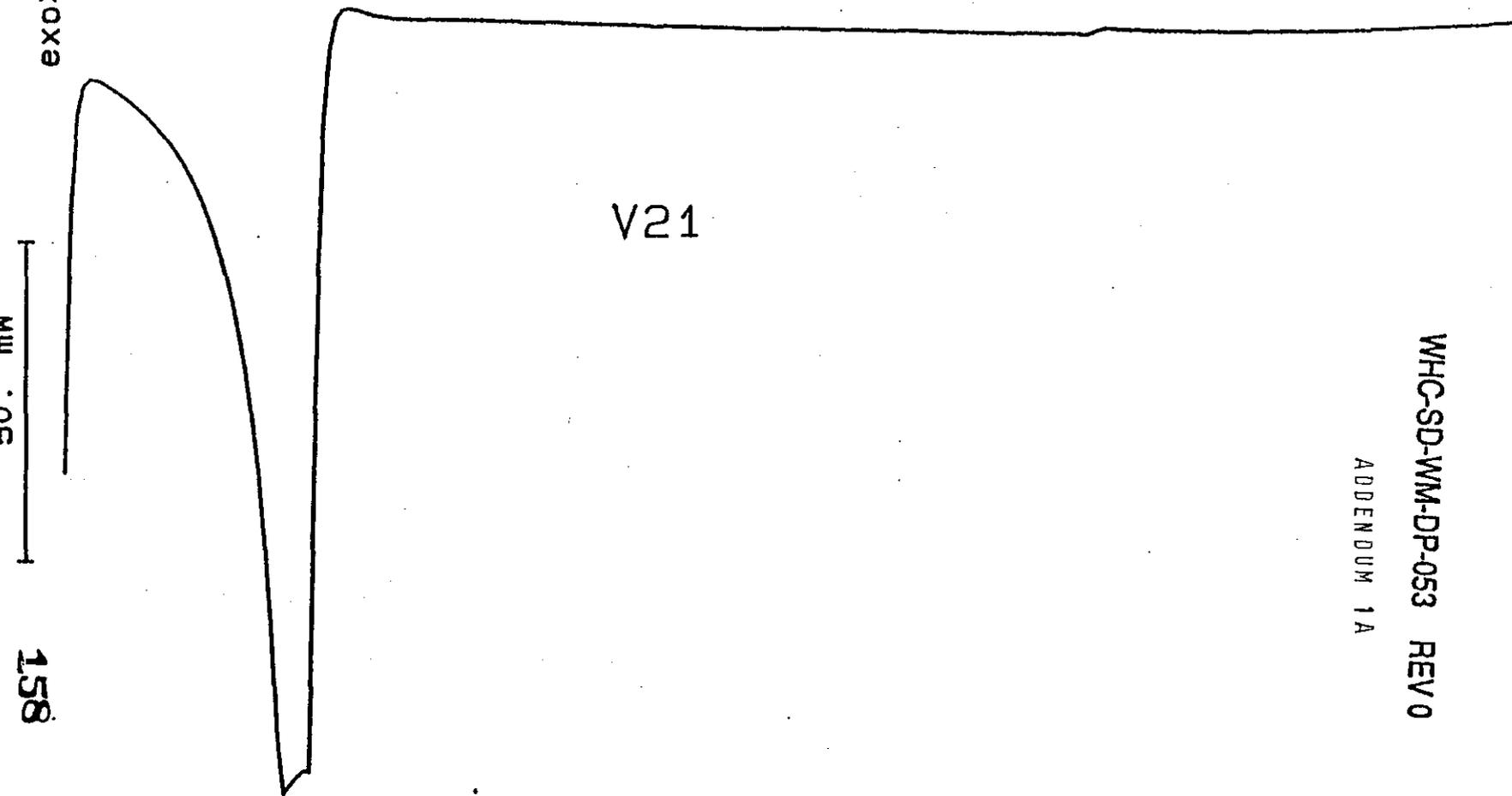
300.

400.

°C

1624

000280



V21 DUP  
10.010 mg

Rate: 10.0 °C/min

File: 00074.001 DSC METTLER 31-Aug-93  
Ident: 222-S Laboratory

<exo>

50.05 mW

159

V21 DUP

WHC-SD-MM-DP-053 REV 0  
ADDENDUM 1A

100.

200.

300.

400.

°C

1A292

000281

DSC STD  
11.339 mg

Rate: 10.0 °C/min

File: 00075.001

Ident:

DSC METTLER

222-S Laboratory

SEPT 1  
31-Aug-93  
RW 9-143

< exo

50.05 mW

160

DSC STD

V-19 *stm*  
11/10/93

Integration

Delta H 2141 mJ

188.8 J/g

Peak 250.6 °C

5.7 mW

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

*Robby Wendland*

0-1-93

100.

200.

300.

400.

°C

1A-293

000282

V23  
9.991 mg

Rate: 10.0 °C/min

File: 00076.001  
Ident:

DSC METTLER  
222-S Laboratory

RW 9-1-93  
~~31-Aug-93~~  
SEPT 1

<exo>

V23

50. mW

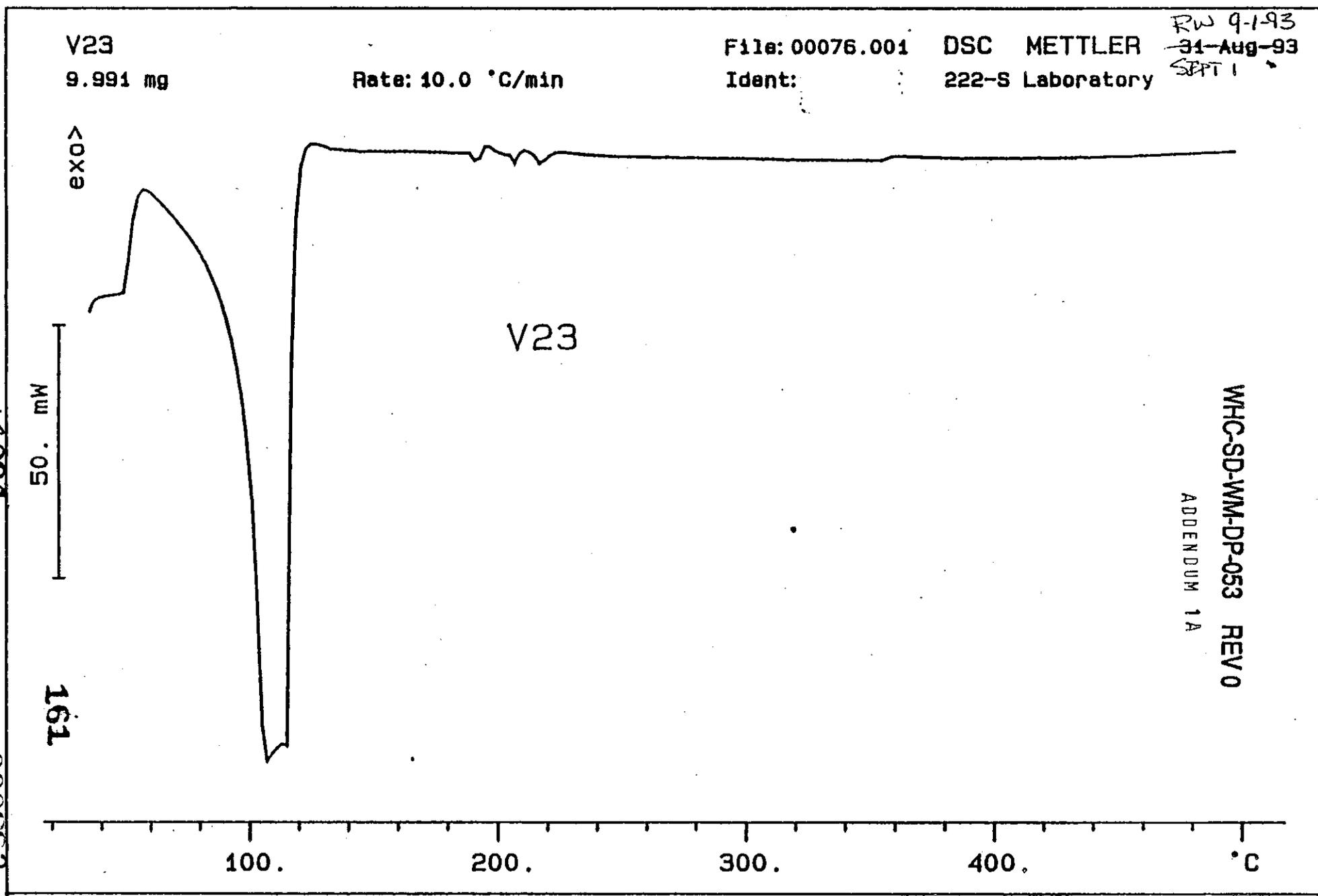
161

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

100. 200. 300. 400. °C

1A-294

000283



V23 DUP  
10.148 mg

Rate: 10.0 °C/min

File: 00077.001

DSC METTLER

01-Sep-93

Ident:

222-S Laboratory

<exo>

50. mW

162

WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

100.

200.

300.

400.

°C

/A-295

000284

DSC STD  
11.601 mg

Rate: 10.0 °C/min

File: 00080.001

Ident:

DSC METTLER  
222-S Laboratory

08-Sep-93

RW 9 993

exo

50 mW

891

Integration  
Delta H 2226 mJ  
191.9 J/g  
Peak 248.5 °C  
6.6 mW

V-24

ADDENDUM 1A

WHC-SD-WM-DP-053 REV 0

100. 200. 300. 400. °C

SIGNATURE ABOVE REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 1128 TO 1129

*Handwritten signature*

11/296

000285

V26

10.147 mg

Rate: 10.0 °C/min

File: 00081.001

Ident:

DSC METTLER

222-S Laboratory

08-Sep-93  
RW 9-9-93

exo >

50 mW

169

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

100.

200.

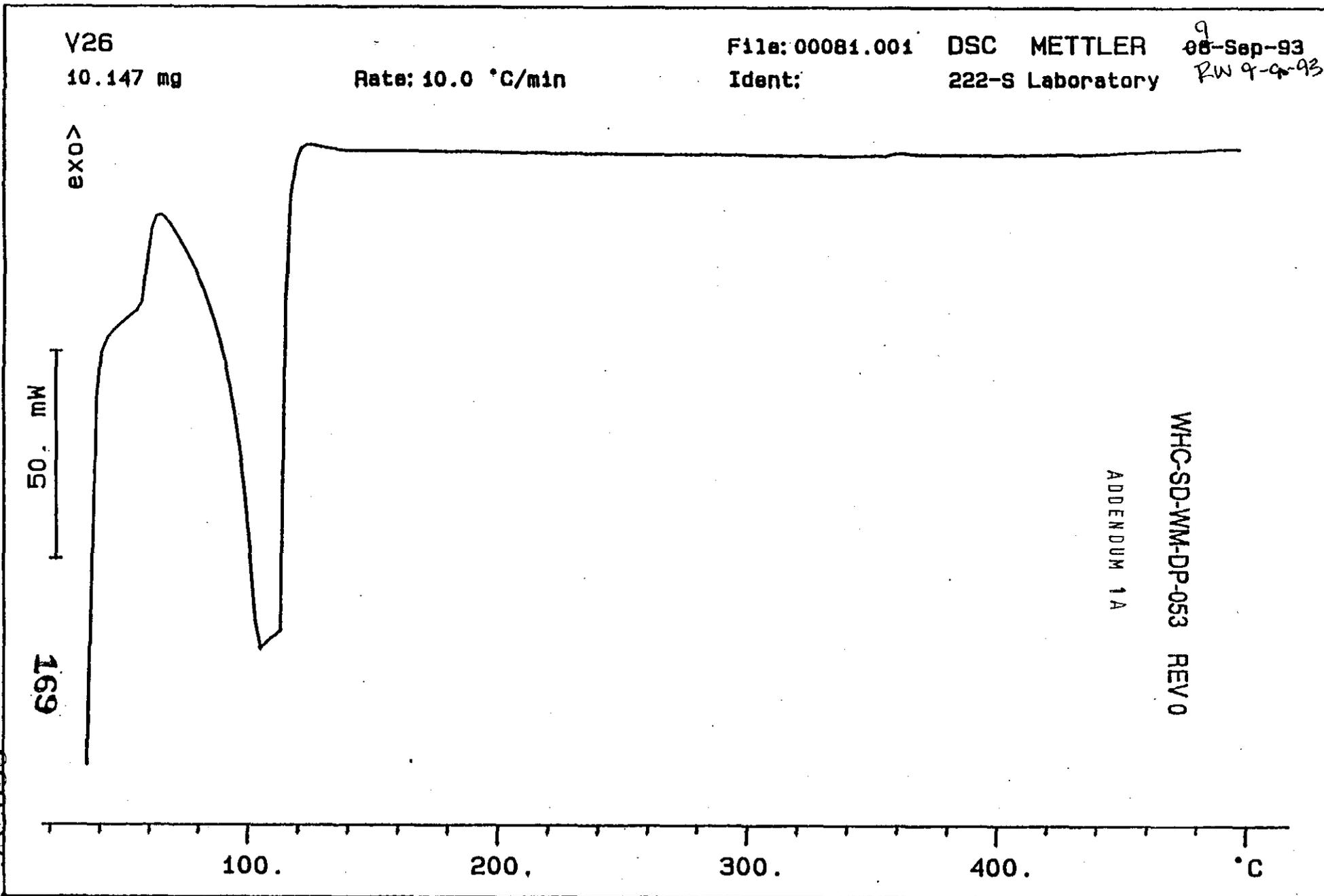
300.

400.

°C

1A-297

000256



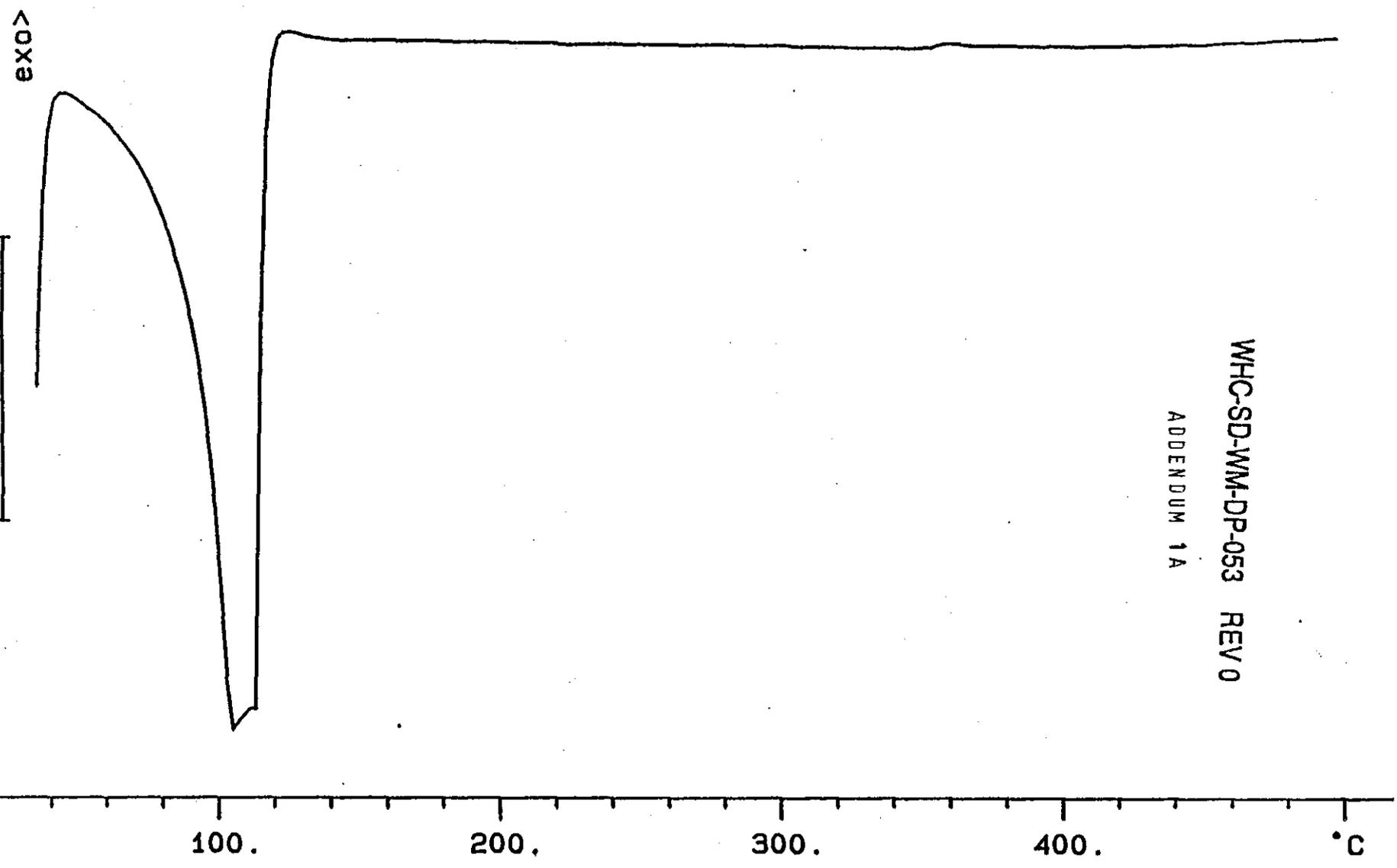
V26 DUP  
10.412 mg

Rate: 10.0 °C/min

File: 00082.001  
Ident:

DSC METTLER  
222-S Laboratory

9  
08-Sep-93  
RW 9-9-93



17A-298

000287

WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

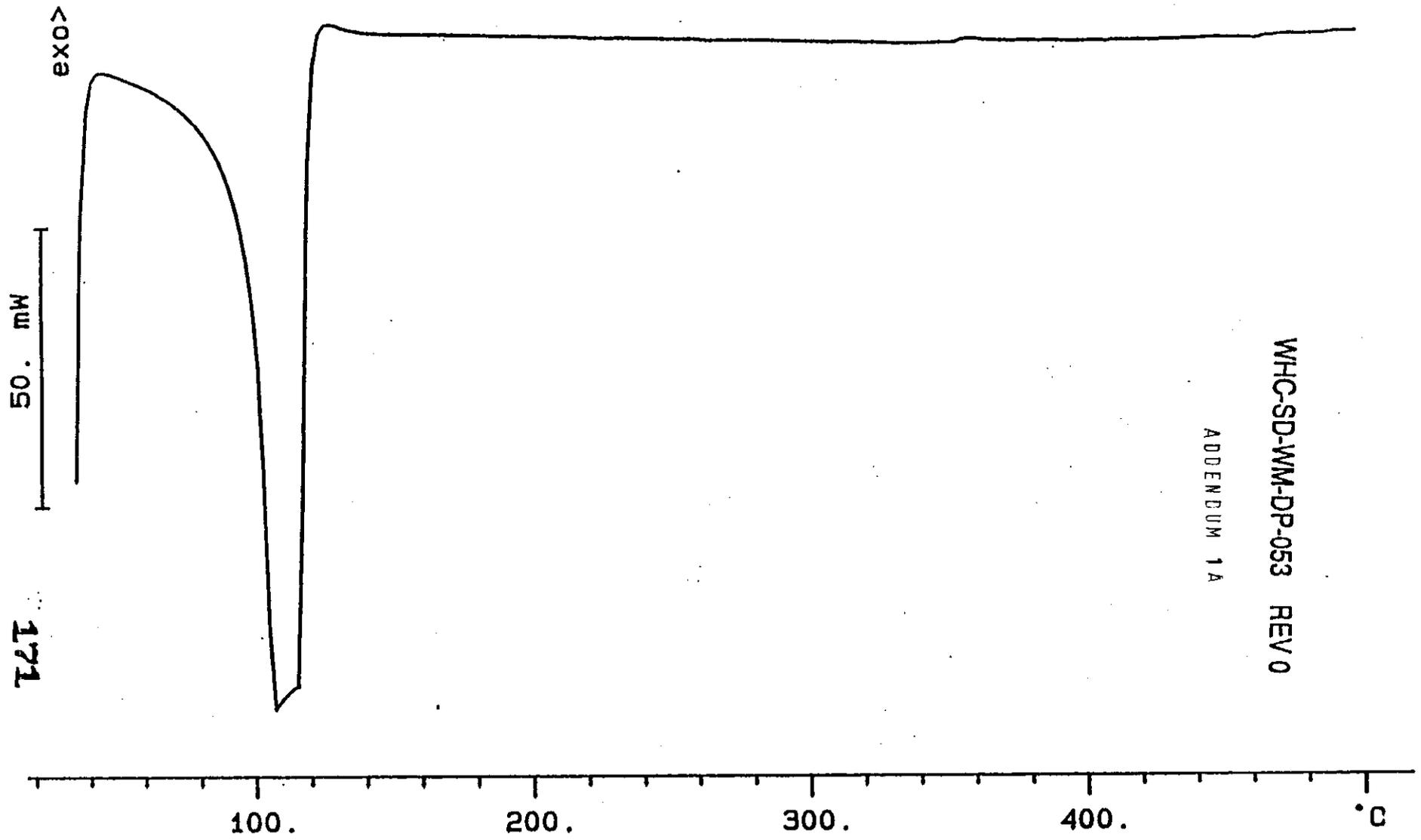
V27  
10.267 mg

Rate: 10.0 °C/min

File: 00083.001  
Ident: :

DSC METTLER  
222-S Laboratory

9  
08-Sep-93  
RW 9-9-93



WHC-SD-WM-DP-053 REV 0  
ADDENDUM 1A

1A-299

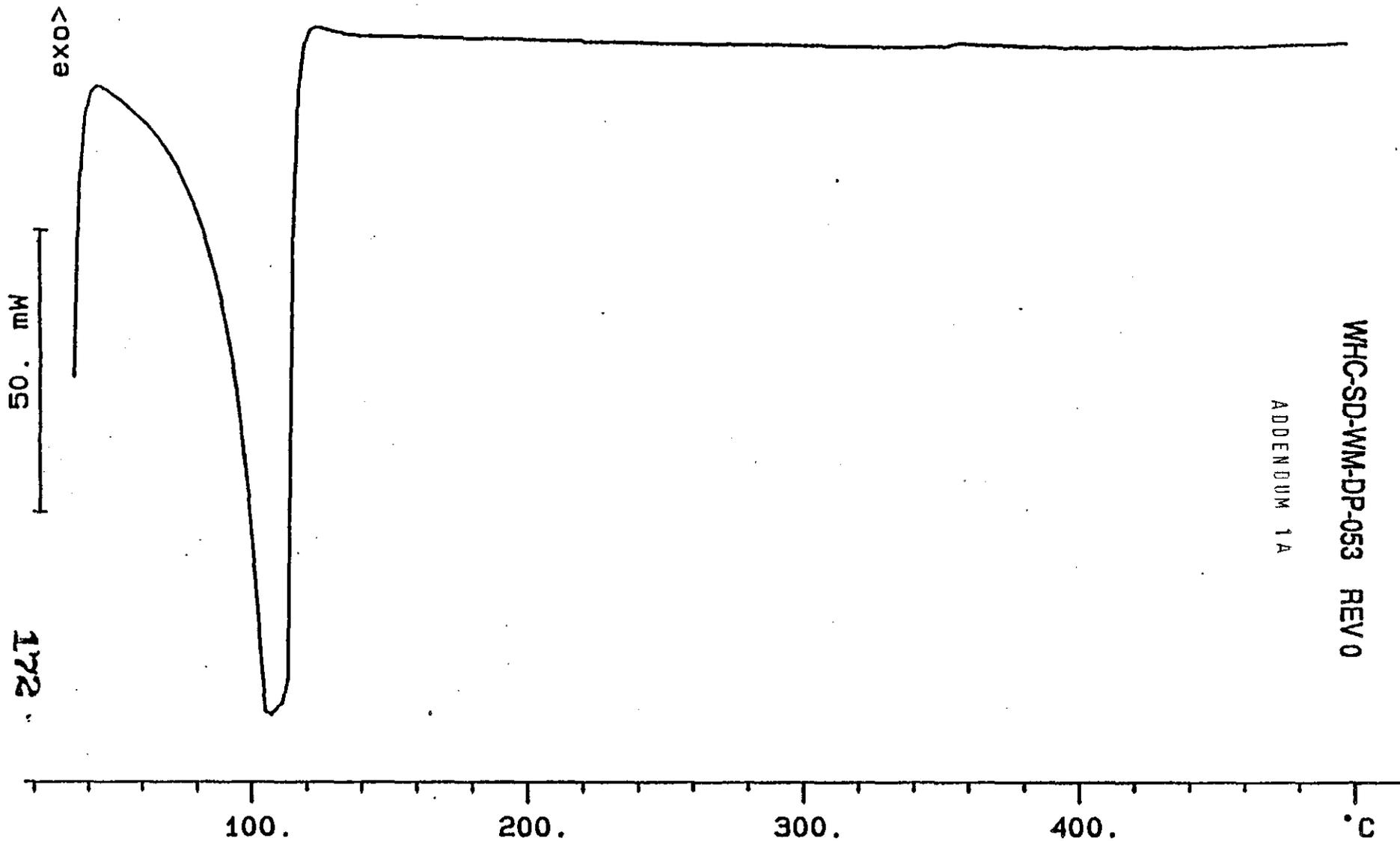
000288

V27 DUP  
10.067 mg

Rate: 10.0 °C/min

File: 00084.001  
Ident:

DSC METTLER 09-Sep-93  
222-S Laboratory



WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

1A-300

000789

V28

10.115 mg

Rate: 10.0 °C/min

File: 00085.001

DSC METTLER 09-Sep-93

Ident:

222-S Laboratory

<exo>

50.05 mW

173

100.

200.

300.

400.

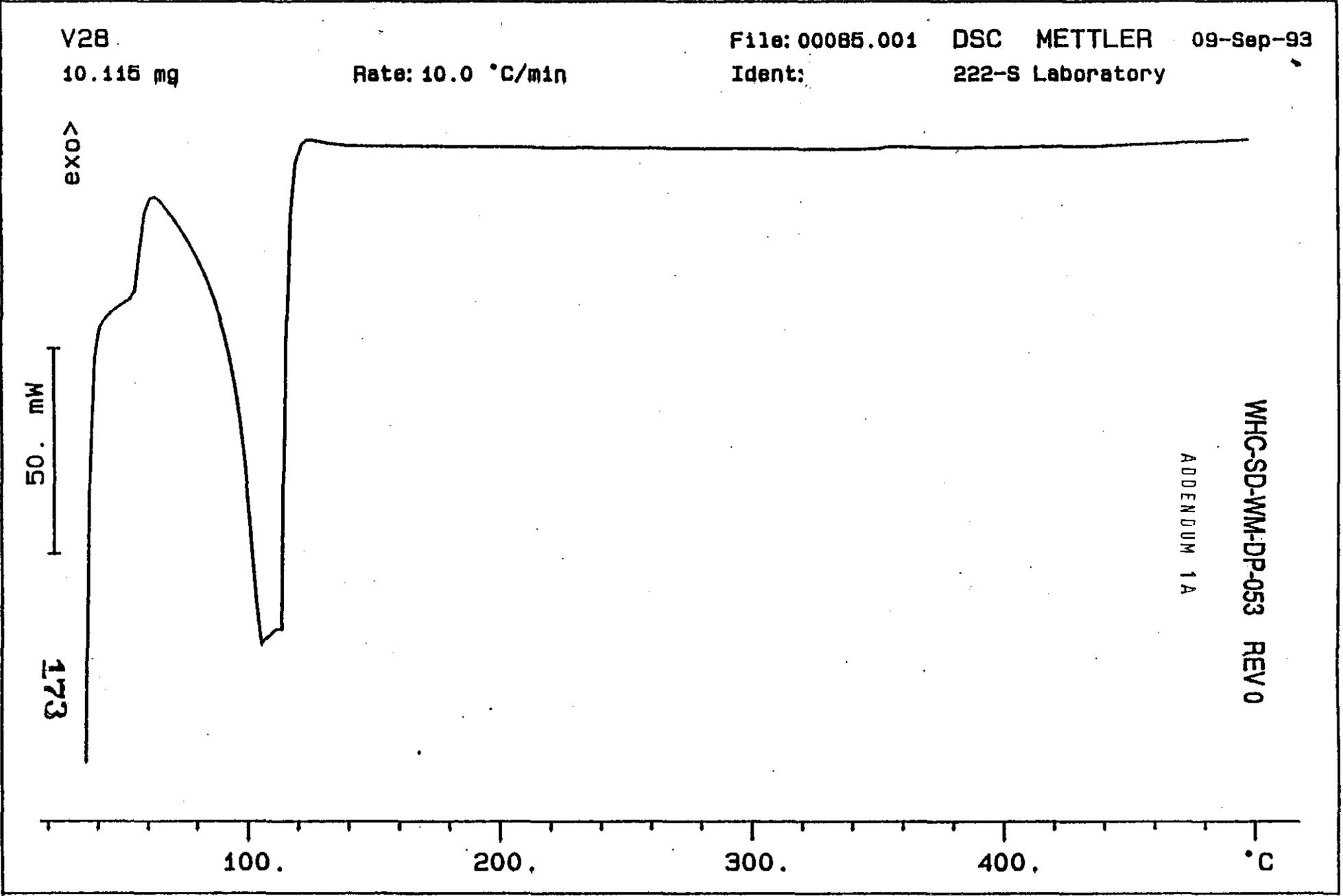
°C

WHC-SD-WM-DP-053 REV 0

ADDENDUM 1A

1A 301

000290



V28 DUP  
10.070 mg

Rate: 10.0 °C/min

File: 00086.001

Ident:

DSC METTLER 09-Sep-93

222-S Laboratory

<exo>

50.05

174

100.

200.

300.

400.

°C

ADDENDUM 1A

WHC-SD-WM-DP-053 REV 0

17302

000091