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ENGINEERING CHANGE NOTICE

Page 1 of 2

1. ECN 600925

Proj. ECN

2. ECN Category (mark one)	3. Originator's Name, Organization, MSIN, and Telephone No.		4. Date
Supplemental []	R. A. St. Denis, PAL, T6-06, 373-1629		12/29/93
Direct Revision [X]	5. Project Title/No./Work Order No.	6. Bldg./Sys./Fac. No.	7. Impact Level
Change ECN []	102-AP Grout Facility Feed Tank	222-S//PAL	4 39
Temporary []			
Standby []			
Supersedure []			
Cancel/Void []	8. Document Numbers Changed by this ECN (includes sheet no. and rev.)	9. Related ECN No(s).	10. Related PO No.
	WHC-SD-WM-DP-046, REV. 0	NA	NA

11a. Modification Work	11b. Work Package No.	11c. Modification Work Complete	11d. Restored to Original Condition (Temp. or Standby ECN only)
[] Yes (fill out Blk. 11b)	NA	NA	NA
[X] No (NA Blks. 11b, 11c, 11d)		Cog. Engineer Signature & Date	Cog. Engineer Signature & Date

12. Description of Change

Changes including, but not limited to, Title, Addition of Summary Statement and Tables, Addition of SW-846 Holding Times, explanations, modifications, clarifications and Addition of units. Title changed to "Characterization Data Package for Grout Facility Feed Tank 102-AP" Case narratives placed in rear of narrative. Exceptions to QC requirements listed. Units Added to Table No. 37.

13a. Justification (mark one)							
		Criteria Change	[]	Design Improvement	[]	Environmental	[X]
As-Found	[]	Facilitate Const.	[]	Const. Error/Omission	[]	Design Error/Omission	[]

13b. Justification Details

Narrative modified in response to customer's request.

14. Distribution (include name, MSIN, and no. of copies)

See Distribution List Attached

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900-013-2 (06/92) GEF095

A-7900-013-1 (06/92)

ENGINEERING CHANGE NOTICE	Page 2 of 2	1. ECN (use no. from pg. 1) ECN-600925

15. Design Verification Required	16. Cost Impact				17. Schedule Impact (days)	
	ENGINEERING		CONSTRUCTION			
[] Yes	Additional	[] $	Additional	[] $	Improvement	[]
[X] No	Savings	[] $	Savings	[] $	Delay	[]

18. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 12. Enter the affected document number in Block 19.

SDD/DD	[]	Seismic/Stress Analysis	[]	Tank Calibration Manual	[]
Functional Design Criteria	[]	Stress/Design Report	[]	Health Physics Procedure	[]
Operating Specification	[]	Interface Control Drawing	[]	Spares Multiple Unit Listing	[]
Criticality Specification	[]	Calibration Procedure	[]	Test Procedures/Specification	[]
Conceptual Design Report	[]	Installation Procedure	[]	Component Index	[]
Equipment Spec.	[]	Maintenance Procedure	[]	ASME Coded Item	[]
Const. Spec.	[]	Engineering Procedure	[]	Human Factor Consideration	[]
Procurement Spec.	[]	Operating Instruction	[]	Computer Software	[]
Vendor Information	[]	Operating Procedure	[]	Electric Circuit Schedule	[]
OM Manual	[]	Operational Safety Requirement	[]	ICRS Procedure	[]
FSAR/SAR	[]	IEFD Drawing	[]	Process Control Manual/Plan	[]
Safety Equipment List	[]	Cell Arrangement Drawing	[]	Process Flow Chart	[]
Radiation Work Permit	[]	Essential Material Specification	[]	Purchase Requisition	[]
Environmental Impact Statement	[]	Fac. Proc. Samp. Schedule	[]		[]
Environmental Report	[]	Inspection Plan	[]		[]
Environmental Permit	[]	Inventory Adjustment Request	[]		[]

19. Other Affected Documents: (NOTE: Documents listed below will not be revised by this ECN.) Signatures below indicate that the signing organization has been notified of other affected documents listed below.

Document Number/Revision	Document Number/Revision	Document Number Revision
NA		

20. Approvals

Signature		Date	Signature	Date
OPERATIONS AND ENGINEERING			ARCHITECT-ENGINEER	
Cog Engineer R. A. St. Denis	Robert St Denis	1-5-94	PE	
Cog. Mgr. J. Kristofzski	[illegible]	1-6-94	QA	
QA J. Langford	J. G. Langford	1-7-94	Safety	
Safety			Design	
Security			Environ.	
Environ.			Other	
Projects/Programs	J M Nguyen	1-10-94		
Tank Waste Remediation System				
Facilities Operations			DEPARTMENT OF ENERGY	
Restoration & Remediation			Signature or Letter No.	
Operations & Support Services				
IRM			ADDITIONAL	
Other				

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INFORMATION RELEASE REQUEST

Reference: WHC-CM-3-4

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[] Brochure/Flier
[] Software/Database
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[] Other

ID Number (include revision, volume, etc.)
WHC-SD-WM-DP-046, REV. 0A

List attachments.
ECN-600925

Date Release Required
12/30/93

Title WHC 222-S LABORATORY GROUT FACILITY FEED TANK 102-AP

Unclassified Category UC-

Impact Level 3Q

New or novel (patentable) subject matter? [X] No [] Yes
If "Yes", has disclosure been submitted by WHC or other company?
[] No [] Yes Disclosure No(s).

Information received from others in confidence, such as proprietary data, trade secrets, and/or inventions?
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Title of Conference or Meeting
NA

Group or Society Sponsoring
NA

Date(s) of Conference or Meeting
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City/State
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Will proceedings be published? [] Yes [X] No
Will material be handed out? [] Yes [X] No

Title of Journal
NA

CHECKLIST FOR SIGNATORIES

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Publication Services	[X]	[]	J.R. Dunphy		1-11-94
Other Program/Project	[]	[X]			

Information conforms to all applicable requirements. The above information is certified to be correct.

	Yes	No
References Available to Intended Audience	[X]	[]
Transmit to DOE-HQ/Office of Scientific and Technical Information	[]	[X]

Author/Requestor (Printed/Signature) Date
R. A. St. Denis R.A. St Denis 1-5-94

Intended Audience
[X] Internal [X] Sponsor [] External

Responsible Manager (Printed/Signature) Date
J. Kristofzski

INFORMATION RELEASE ADMINISTRATION APPROVAL STAMP

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Date Cancelled | Date Disapproved

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Part 1

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SUPPORTING DOCUMENT

1. Total Pages ~~60~~ 5.921

2. Title	3. Number	4. Rev No.
102-AP Grout Facility Feed Tank	WHC-SD-WM-DP-046	0A

5. Key Words

102AP, Grout Feed Tank

6. Author

Name: R. A. St. Denis

R. A. St Denis 1-5-94

Signature

Organization/Charge Code 124J0/J120H

7. Abstract

NA

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RECORD OF REVISION	(1) Document Number WHC-SD-WM-DP-046	Page 1

(2) Title
102-AP GROUT FACILITY FEED TANK

CHANGE CONTROL RECORD

(3) Revision	(4) Description of Change - Replace, Add, and Delete Pages	Authorized for Release (5) Cog. Engr.	(6) Cog. Mgr. Date
0	(7) EDT-140712	R. St. Denis	J. Kristofzski 1/11/94
0A RS	Pages vii, and 2 through 59.1 have been revised and in some cases rearranged.	R. St. Denis	J. Kristofzski 1/4/94

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Total Organic Carbon G341, G342 3589
Total Organic Carbon G348, G349 3600
Total Organic Carbon G352, G353 3610
Total Organic Carbon G358, G359 3621
Total Organic Carbon G363, G364 3632
Total Organic Carbon G363 3643
Total Organic Carbon G364 3651
Total Organic Carbon G443 3659
Total Organic Carbon G459, G460 3670
Total Organic Carbon G470, G471 3682
Total Organic Carbon G476, G477 3696

Total Inorganic Carbon G333, G338 3710
Total Inorganic Carbon G341, G342 3724
Total Inorganic Carbon G348, G349 3738
Total Inorganic Carbon G352, G353 3752
Total Inorganic Carbon G353 3766
Total Inorganic Carbon G353 3776
Total Inorganic Carbon G358, G359 3786
Total Inorganic Carbon G363, G364 3800
Total Inorganic Carbon G459, 460 3815
Total Inorganic Carbon G460 3829
Total Inorganic Carbon G470, G471 3839
Total Inorganic Carbon G476, G477 3853

This report consists of pages 1 though 3866 plus 59.1, 2917.1 through 2917.10 and 3609.1, also pages 7, 30, and 125 to 144 were intentionally left blank.

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Introduction

On 4/30/93 grout feed tank 241-AP-102 was sampled for a full characterization under the protocol listed in Hanford Grout Disposal Program-Campaign 102 Feed Characterization and Test Plan, WHC-SD-WM-TP-136, and Technical Project Plan for The 222-S Laboratory in Support of The Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP, and 102-AP, WHC-SD-WM-TPP-008.

The analyses in this data package were performed by the Westinghouse Hanford 222-S Laboratory under the following three documents: 1) "Hanford Grout Disposal Program Campaign 102 feed Characterization and Test Plan" (WHC-SD-WM-TP-136, Revision 0), 2) "Grout Treatment Facility Characterization Project, Fiscal Year 1993, Statement of Work For The Processing and Analytical Laboratories" (WHC-SOW-92-005, Revision 1), and 3) "Technical Project Plan For the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP, and 102-AP" (WHC-SD-WM-TPP-008, Revision 0). These documents will hereafter be referred to as the 1) FCP, 2) SOW, and 3) TPP respectively.

Laboratory operations at the 222-S 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), unless superseded by the FCP, the SOW of the TPP. Deviations from these guidelines are documented in letters of instruction from Grout Technology, Engineering Change Notices (ECNs) and in this narrative.

Tank 241-AP-102 (102-AP) are to be prepared as feed wastes for processing and disposal during campaign 102 of the Hanford Grout Disposal Program (HGDP). This campaign is scheduled to be initiated during October 1993. A historical profile of 102-AP is as follows:

Tank 102-AP is a 1,140,000 gallon radioactive waste tank used to composite solutions from several sources, and mix them prior to blending with dry grout material. After the last grout campaign (campaign 101) in which grout was blended and poured into vault 218-E-16-101 (vault 101), all of the contents of tank 102-AP were converted to grout except for a residual "heel" of liquid. The leachate and excess drainable liquids (that result from the curing process of grout) from that campaign were returned back to 102-AP and combined with the heel. Wastes from the Plutonium Uranium Extraction facility, consisting of neutralizing agents, were then added to 102-AP, as was solution from another radioactive waste storage tank (241-AN-106), which was characterized prior to transfer of the solution (Welsh, 1991).

ANALYTES	MEAN CONCENTRATION (mg/L or Ci/L)	ANALYTICAL RELATIVE STANDARD DEVIATION (%)
Np-237	< 4.65E-07	12.7*
Am-241	4.19E-07	23.9
Sr-90	1.44E-03	6.9
I-129	< 3.71E-08	3.9*
H-3 ***	1.30E-05	76.0

* Relative Standard Deviation is based on results from standards.

** Relative Standard Deviation can not be calculated because no data are available either from samples nor standards.

*** Tritium results are suspect due to unacceptably high spike recovery (1369%). This is probably due to the limitation of the analytical method.

The agitation and heating of the waste prior to sampling, the locations and large number of samples, the use of accepted sampling technique assure a high level of certainty that the data are representative of the waste in the tank.

Calibration standards, reference samples (method standards), matrix spike and matrix spike duplicates, and surrogate samples were employed, as appropriate, to assure that the accuracy of the data are acceptable for the anticipated data usage.

Field and laboratory duplicates and an appropriate number of representative samples assure the precision of the data are acceptable for the anticipated data usage.

ANALYTES	MEAN CONCENTRATION (mg/L or Ci/L)	ANALYTICAL RELATIVE STANDARD DEVIATION (%)
F^-	< 2.09E+02	4.0*
Cl^-	2.72E+03	5.6
NO_2^-	3.58E+04	5.8
NO_3^-	7.58E+04	5.7
PO_4^{3-}	1.16E+04	7.5
SO_4^{2-}	4.51E+03	5.8
OH^-	9.15E+03	1.7
Percent Water	75.00 %	0.3
SpG	1.20	0.2
CO_3	2.67E+04	3.9
Total Organic Carbon	3.28E+03	3.8
CN^-	2.46E+01	1.3
NH_4	< 1.60E+02	4.2
Cs-137	2.28E-01	3.4
Cs-134	< 5.68E-05	**
Co-60	< 8.09E-01	2.3*
Ce/Pr-144	< 1.36E-03	**
Sb-125	< 7.35E-04	**
Ru/Rh-106	< 2.52E-03	**
Nb-94	< 4.62E-05	**
Pu-239	< 7.48E-08	5.3*
Pu-238	< 1.63E-07	**
C-14	4.99E-07	15.3
Tc-99	8.56E-05	5.5

TANK 241-AP-102

CASE NARRATIVE

Characterization Data Package
for the Grout Facility Feed Tank 241-102-AP
SUMMARY

This data package contains the "raw" data from the characterization of Tank 102-AP waste samples taken in April 1993. A summary of the inorganic and radiochemical data are provided in Table 1. Data from the organic analyses performed by the Pacific Northwest Laboratory are provided in a separate package (PNL 9005). Both data packages have been validated by the Hanford Analytical Services Management. The results of a statistical analysis of the data and a comparison to the Grout Treatment Facility feed acceptance criteria are provided in Welsh (1993).

Table 1. Summary of Tank 102-AP Waste Characterization Data--Inorganic and Radiochemical Analyses

ANALYTES	MEAN CONCENTRATION (mg/L or Ci/L)	ANALYTICAL RELATIVE STANDARD DEVIATION (%)
Al	1.16E+04	1.2
Sb	< 9.47E+00	2.5*
Ba	2.84E-01	16.1
Be	1.46E-01	6.6
Cd	1.47E+00	6.7
Cr	6.18E+02	1.8
Fe	3.73E+00	16.5
Pb	< 5.33E+00	1.7*
Ni	2.66E+01	2.1
K	1.29E+03	1.9
Ag	< 1.25E-01	4.4*
Na	1.02E+05	2.3
P	3.06E+03	2.7
Se	3.67E-01	4.5
U	4.53E+00	42.0
As	< 1.02E-01	11.1*
Hg	< 5.00E-03	4.4*

Sampling

Tank 102-AP characterization analyzed samples originating from 3 fixed, vertical 4 inch risers, located 120 degrees apart on a 20 foot radius.Samples were taken via bottle-on-a-string method (Fig. 1).

A total of twenty-five samples were obtained from 102-AP for analysis. Seven (two from each riser, plus one field duplicate) were sent to Battelle's Pacific Northwest Laboratory (PNL) for organic compound characterization. The remaining eighteen samples were sent to the Westinghouse Process and Analytical Laboratories (PAL) for inorganic and radiochemical characterization. Of those eighteen samples, six were intended for only limited characterization (Cesium, Phosphate, and Sodium), to determine whether the tank was homogenous. The other twelve (three samples from each riser, plus a field duplicate from each riser) were intended for full characterization.

A composite solution was prepared from 15 of the eighteen samples (field duplicates were not included in the composite preparation), by mixing varying volumes of each sample together. The Proper volume ratios to use for each sample were based on the homogeneity test results. Since the samples were determined to be from a homogeneous, non-layered solution, equal volumes of each of the 15 samples were used to prepare the composite.

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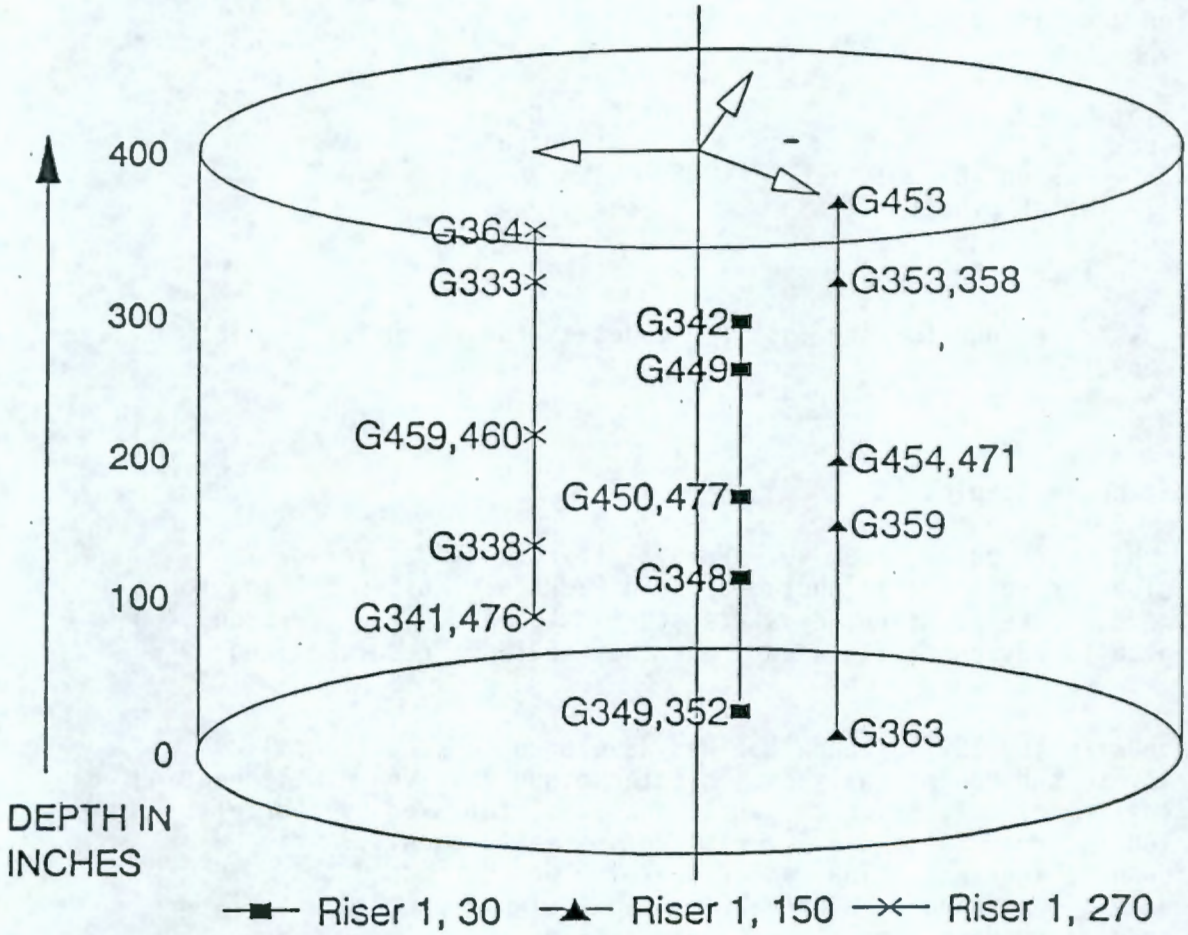
Figure 1. Tank 241-AP-102 Sample Locations



Sample Tracking and Laboratory Identification

The laboratory tracks samples through a laboratory identification number (to identify the sample), followed by an extension that identifies the method of preparation (direct, acid digest, fusion, etc), sample type (standard, blank, duplicate, or spike), and subsequent analysis (IC, ICP, GEA, etc.). The laboratory ID begins with a letter designator to identify the program; "G" identifies that the sample is associated with grout. A numerical sample number follows (e.g. G327). The extension is typically comprised of four numbers. The first number represents the preparation method type; The second is sample type, and the last two designate the type of analysis performed. The meanings of the first two numbers in the extension are the most useful in interpreting the report*. The cross reference for the first two numbers in the extension are listed below:

1ST Number	2nd Number
5 - Direct	5 - Standard
6 - Fusion Dissolution	6 - Blank
7 - Water Digest	7 - Sample
8 - Acid Digest	8 - Duplicate
9 - TCLP Prep	9 - Spike

* - The cross reference for the last two codes is too extensive to list in this report.

Solids Formation in Samples

The samples that were pulled from 102-AP initially existed as yellow solutions, clear of solids (See photographs in Package). After sitting for a few days at ambient temperature, crystals started to form. The TPP predicted that certain salts may precipitate out, but they could be resolubilized by heating.

In compliance with the TPP, a technique was developed to keep the solids in solution prior to subsequent analysis. This technique involved gentle heating of the samples immediately prior to sample analysis, followed by a brief cooling period in order to obtain accurate volume measurements. This procedure for handling the solids, unfortunately, was not adequately communicated to all working within the PAL, and a significant amount of analyses were performed on samples containing solids.

Upon discovery of this, all lab work was suspended until an adequate recovery plan could be developed. A recovery process was implemented as follows:

1) Each analytical method was reviewed to determine which ones allowed for solids to be dissolved as part of the procedural steps. It was found that only ICP and AA methods employed an acid digest (heating/dilution) preparation step, to adequately solubilized any solids prior to analysis.

1) The compound was analyzed to determine what elements it was comprised of.

2) All samples were reanalyze for those elements that comprised the solid compound.

3) Four samples were picked at random to reanalyze for all the full protocol of elements. The only elements that were omitted were those where the analytical method employed a digestion/heating step. This was performed in order to demonstrate that no elements other than those found comprising the solid were carried down or co-precipitated with the solid.

The precipitate was analyzed by the PAL using polarized light microscopy. The crystal structure and birefringence (difference in refractive indexes in the x and Y direction) was indicative of hydrated sodium phosphate.

The solid contained sodium phosphate, therefore it was concluded that as long as no other constituents coprecipitated, they were the only analytes that needed to be redetermined. Sodium is determined by ICP, therefore it was not reanalyzed for. Phosphorus was reanalyzed for by obtaining a new (heated aliquot) from each parent sample, and submitting it for IC analysis. Special handling instructions accompanied the new aliquots to prevent the reformation of solids. To determine if any other anions were carried down with the Na3PO4, four of the above samples submitted for IC analyses were used to reanalyzed for all constituents in the full grout characterization protocol. Sample numbers for these heated aliquots, and their parent sample are listed in Table 4.

Table 2. Laboratory Tracking Numbers assigned to Sample aliquots that Utilized Special Solids Dissolution Methods.

Original Sample Numbers	New Aliquot Numbers
G299	G509
G301	G523
G302	G477*
G303	G510
G305	G511
G306	G521
G307	G527
G308	G522
G309	G515
G310	G471*
G311	G516
G313	G533
G315	G534
G317	G528
G318	G517
G319	G536
G322	G529
G323	G476*
G327	G470*

* - THESE SAMPLES WILL BE ANALYZED FOR THE ENTIRE PROTOCOL OF ANALYTES TO VERIFY THAT THE SODIUM PHOSPHATE PRECIPITATION DIDN'T CO-PRECIPITATE OTHER ANALYTES.

Results of the anion on four sample aliquots showed a significant increase in the concentration of phosphate over previous results. All other anions concentrations were essentially the same as the samples with solids where the analytical aliquot was taken from the supernate. In addition, GEA values were essentially the same. These facts, together with process knowledge and expert opinion, indicated that precipitation seemed to occur without co-precipitating more soluble constituents. Consequently, a repeat of all analyses on the four samples was performed to determine whether resolubilized results were comparable to those where only an aliquot of the supernate was tested. Both sets of data are comparable therefore the data in question are considered valid.

Laboratory Operations

An Analytical Batch Summary Sheet (ABSS) appears with the analytical batches. The ABSS does not show any raw data. It summarizes the calculated results for samples, duplicates, spikes, blanks, averages RPD's, and detection limits from the chemist's initial data evaluation, and provides a means of obtaining descriptive results from the chemist and chemical technologist on each sample batch. While not required as part of the final package, these forms aided in preparing this narrative and provided insight into some of the problems encountered during sample analysis.

forms aided in preparing this narrative and provided insight into some of the problems encountered during sample analysis.

Quality Control

The required QC criteria are listed in Appendix A of the TPP. The requirements are summarized as follows:

- Matrix Spikes
  - Frequency: one per tank or each unique matrix for each analyte.
  - Criteria: 75-125%, spike must be >25% sample concentration.
  - Corrective Action: one rerun and narrative

- Laboratory Control Standard
  - Frequency: ICP and AA metals: One digested and one undigested standard before each batch is analyzed and one digested and undigested standard after each batch is analyzed.

    One undigested standard per batch for all remaining analytes.
  - Criteria: LMCS control limit (usually 3σ of history values) Digested standards are not governed by QC requirements, but are used as a troubleshooting aid.
  - Corrective Action: stop and fix; otherwise, explain in narrative

- Duplicates

  - Frequency: 100% duplicates prepared from separate dilutions (and/or digestions) of the original sample.

  - Criteria: Initial criteria for rerun is when the RPD exceeds the measurement error for LMCS standards (3σ of history values for LMCS standard recovery). After all samples have been analyzed, the rejected data is reevaluated against 3σ as determined from the sample population. Both criteria assumes sample >10 times the DL. The TPP stated that neptunium and Iodine 129 methods are normally biased 20-30 percent low, and may not always meet the requirement.

  - Corrective Action: One rerun if initial criteria fails; Reevaluation of the initial, rejected data against final criteria; explain in narrative if RPD fails final criteria.

- Preparation Blanks

  - Frequency: one per preparation batch

  - Criteria: <20% sample result or higher than normal

  - Corrective Action: identify problem; rerun batch if necessary; explain in narrative.

The exceptions to the above requirements are as follows:

- ^{90}Sr, ^{14}C, ^{3}H, ^{99}Tc, ^{129}I, $^{239/240}Pu$, ^{241}Am have a spike, tracer, or carrier added to each sample; no additional matrix spikes were required by the technical project plan, however some were performed at the request of the project coordinator.

- GEA analyses are not affected by sample matrix effects, therefore no spikes will be performed on GEA samples.

- Specific gravity (SpG), GEA, OH and pH methods do not require a spike.

- Percent water and acid digest procedures do not employ a blank.

- ICP, hydride atomic absorption spectrophotometry (HYAA), cold vapor atomic absorption spectrophotometry (CVAA), and ion chromatography (IC) require additional, method-specific QC. Instrument calibration and check standards are run according to specific procedure protocols.

For all sample analyses are repeated at least once if the spike recovery is outside of 100% ±25%, (provided the spiked sample concentration is at least 25% greater than the sample concentration); or when the duplicates have a relative percent difference (RPD) greater than 3σ of the LMCS historical standard recovery (provided the analytical results are greater than 10 times

the instrument/method DL). If the rerun results did not meet the QC criteria and the chemist had no explanation or "fix" then additional reruns were not required. This was the case in the tritium analyses. These situations were described in this narrative. The entire batch did not have to be rerun unless the standard or blank for the batch failed.

For ICP analysis the following criteria were used when evaluating whether or not to rerun an analysis:

- **Initial calibration verification standard (ICV), continuing calibration verification standard (CCV)** — Recovery 100% ±10% If >10 elements are out on any single standard, the whole batch was rerun.

- **LMCS (Undigested) Standard** — Both a beginning standard and an ending standard must be performed. Recoveries must be 100% ± 3σ of LMCS historical value.

- **Duplicates** — Initial criteria for rerun is when the RPD exceeds the measurement error for LMCS standards (3σ of history values for LMCS standard recovery). After all samples have been analyzed, the rejected data is reevaluated against 3σ as determined from the sample population. Both criteria assumes sample >10 times the D.L.. Exceptions were noted and evaluated for rerun based on the type of metal, blank contamination, and standard performance.

- **Spikes** — 100% ±25% recovery if they are at least 25% of the analyte concentration.

- **Serial Dilutions** — ≤10% difference for those metals >1,000 μg/g. Exceptions were noted.

- **Preparation Blanks** — Note unusual results.

Results of ICP evaluations will either be found in the batch narrative, this narrative or inferred due to the presence of a rerun result.

Holding Times

Tank 241-AP-102 was sampled on 04/30/93. The last sample was received into the PAL on 05/01/93. The acid digestion for the ICP individual samples was finished on 05/20/93. The ICP metal analyses for the individual samples were completed on 07/15/93. The composite sample (G443) was prepared on 05/14/93. The Acid digestion (ICP prep.) was completed on 05/18/93. The ICP metal analysis for the composite was completed on 07/01/93.

Hg analyses for the composite sample were completed on 06/30/93. The TPP did not require Hg to be determined for the individual samples.

HOLDING TIMES

ANALYTE	SW-846 REQUIREMENT
CHLORIDE	28 DAYS
NITRATE	48 HRS.
SULPHATE	28 DAYS

ANALYTE	SW-846 REQUIREMENT
CHROMIUM VI	24 HRS.
MERCURY	28 DAYS
OTHER METALS	180 DAYS

ANALYTE	SW-846 REQUIREMENT
TOC	28 DAYS

ANALYTE	SW-846 REQUIREMENT
RAD. CHEM. (alpha, Beta, etc)	180 DAYS

ANALYTE	SW-846 REQUIREMENT
TCLP (Cr)	24 HOURS

102-AP Results

Analytical results for 102-AP appear in the master summary sheets. The summaries were generated as Lotus[1] version 3.1+ spreadsheets which were intended to aid in review of analytical data, and may not include all of the associated laboratory control standards, dates, or reflect the proper number of significant figures. The summary spreadsheets also will not show the tracer or carrier values that were used to determine radiochemical results. Tracer and carrier recoveries were not calculated separately, but were indirectly integrated into the equations used to calculate sample results. The spreadsheet summaries display analytical results, blank data, detection limits (DLs), LMCS recoveries, spike recoveries, averages for duplicates, and relative percent difference (RPD) between duplicates. When a spiked sample analysis was performed, that data is included with the other results for that sample. When values were found to be less than detection limit, "< D.L." was listed. Tables, spreadsheets, and figures are used throughout this report to show various comparisons and to display mass and charge balance results.

Detection Limits

Detection limits listed are method detection limits. They typically represent instrument detection limits that have been corrected for the dilution factor employed in the method. Not all samples require the same dilution factor in order to get within the calibration range of the instrument. For this reason, it is not abnormal to see two different detection limits listed for the same analyte, and the same method.

For example, if a 499 ppm sample is diluted by 10 (50 ppm), and analyzed on an instrument that has a detection limit of 50ppm, the only thing that can be concluded is that the result of the dilution was < 50 ppm. The most we could say about the sample (since it was 10 times more concentrated) is that it had less than 500 ppm. If the same sample were to have been instead diluted by 100 (4.99 ppm) prior to analysis on the same instrument, the result of the dilution would have still been < 50 ppm, but the most we could say about the undiluted sample was that it was 100 timed the detection limit, or < 5000 ppm.

Some instrument detection limits require a discrete quantity (expressed as a weight value) in order to be measured. Detection limits for these methods were determined by dividing that discrete quantity through by the volume of blank (or sample) used in the analysis. This "minimum concentration" was then corrected for any dilutions made to the sample (or blank) prior to analysis.

With Radiochemistry, each measured quantity, be it a blank, or a sample, has its own unique detection limit that is calculated based on the measured background present for the sample, and the time of the count. This value is then adjusted for sample size and dilution factor.

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[1] Lotus is a trademark of Lotus Development Corporation.

Blanks

Blanks were either reagent blanks or method blanks. A method blank was one that was carried through each step of the procedure (extraction, precipitation, etc.). A reagent blank was used only as a diluent for the sample. Regardless of which type of blank was analyzed, it was corrected for sample dilution in order to determine how much of final result may have been contributed by the blank.

For example, say the instrument response gave 5 for the blank and 50 for a sample that had been diluted by 100. Assuming that instrument response was directly proportional to the analyte concentration, then 10 percent of the sample concentration (5 out of 50) may have been from the blank (reagent) contribution. When the 100X sample dilution factor was then applied to the sample concentration, it had to also be applied to the blank concentration in order to keep the blank concentration at 10% of the sample concentration. This allowed direct comparison of the background and degree of contamination present in the method to the sample results.

When a sample result failed the QC criteria the results of both the original and the rerun were reported. The results were presented in chronological order for that analyte. In most cases the presence of multiple results for the same sample point indicates that there was a QC failure, and no single set of results met the QC criteria sufficiently well to stand independently.

Direct internal letters and informal memos were used by the customer to communicate minor deviations from the TPP and the Feed characterization plan. A miscellaneous correspondence section contains these memos and letters, and is located in the summary section. Major deviations were documented in engineering change notices.

Several other groups within the Processing and Analytical Laboratories besides the 222-S group produced analytical results and reports for the 102-AP samples. Personnel from the Process Chemistry (PCL) and Special Studies groups also participated in this effort.

PNL Samples

Six samples and a field duplicates were sent to PNL for Organic Characterization. Table 5 lists these samples and their PAL-generated Shipping Numbers.

Table 3. 102-AP Samples Sent to PNL for Analysis

Shipping Number	Location (riser, height)
G300	30°, 306"
G304	30°, 123"
G312	150°, 162"
G314	150°, 18"
G316	270°, 372"
G320	270°, 226"
G321 (Field Dup.)	270°, 226"

SECTION I. RECEIPT AND SAMPLE DATA

RECEIPT

A series of laboratory identification numbers were initially created for the eighteen 125 ml samples received from grout tank farms, and the composite sample. Aliquots pulled from the original samples for laboratory analyses were labeled with a different laboratory identification number. All additional aliquots pulled from the original samples were labeled with new, unique laboratory identification numbers if they were to be stored or treated differently than the original aliquot submitted to the laboratory.

Table 6 lists the sample numbers assigned to the 102-AP samples as recorded on the chain-of-custody record for core sampling. It also contains a Cross reference listing the original sample/aliquot numbers, subsequent aliquot identification numbers, the month and year of the sampling and the riser location and depth from which the sample was pulled.

Table 4. 222-S Chain-of-Custody Samples Numbers

Laboratory Shipping (COC) ID	Original[1] Lab. Sample Aliquot ID	New[2] Lab. Sample Aliquot ID	Tank Location	Date Sampled
G299	G342	G509[3]	30°, 306"	04/28/93
G301	G449	G523[3]	30°, 271"	04/28/93
G302	G450	G477[4]	30°, 182"	04/28/93
G303	G348	G510[3]	30°, 125"	04/28/93
G305	G349	G511[3]	30°, 34"	04/28/93
G306 (DUP)	G352	G521[3]	30°, 34"	04/28/93
G307	G453	G527[3]	150°, 391"	04/29/93
G308	G353	G522[3]	150°, 334"	04/29/93
G309 (DUP)	G358	G515[3]	150°, 334"	04/29/93
G310	G454	G471[4]	150°, 208"	04/29/93
G311	G359	G516[3]	150°, 162"	04/29/93
G313	G363	G533[3]	150°, 18"	04/29/93
G315	G364	G534[3]	270°, 372"	04/30/93
G317	G333	G528[3]	270°, 334"	04/30/93
G318	G459	G517[3]	270°, 226"	04/30/93
G319 (DUP)	G460	G536[3]	270°, 226"	04/30/93
G322	G338	G529[3]	270°, 148"	04/30/93
G323	G341	G476[4]	270°, 99"	04/30/93
G327	G443	G470[4]	COMPOSITE	N/A

1- All aliquots contained solids. All analyses were performed on supernate except for ICP, which was performed on a digestion portion of the aliquot before solids had formed.

2- Aliquots were heated to keep solids in solution. Samples were briefly cooled to ambient temperature prior to analyzing.

3 - These samples were analyzed by ion chromatography only, in order to reanalyze for phosphate after the sodium phosphate solids were dissolved.

4 - These samples were analyzed for the entire protocol of analytes to verify that the sodium phosphate precipitation didn't pull down other analytes.

VISUAL

Each 125 ml Sample was a clear yellowish liquid when taken from the tank. At the time of sampling there were no solids. The samples were stored at ambient outside temperature for the first 24 hours. Within a 24 hour period of time after the samples were taken, a white precipitate had formed in the bottom of each sample bottle. The samples were transferred to a warmer environment (ambient temperature inside of the 222-S laboratory), but the solids persisted.

SAMPLES AND SUBSAMPLES FOR 102-AP

Subsamples

Table 7 contains a cross reference of the serial numbers used by the 222-S Laboratory for tracking sample analyses, the analytes requested for each sample, and the applicable RSA. The RSA is the form by which an analyte list is defined for a sample. All sample numbers have an "G" prefix assigned to them. Samples, duplicate samples, and spiked samples for a particular sample point and analysis all had a single serial number.

Table 5. 102-AP Laboratory Sample Identification and Analyte List.

Orig. Sample Shipping No. (Laboratory Aliquot No.)	RSA Number	Analytical Method
Full Characterization (W/Undissolved Solids)		
G299 (G342) G303 (G348) G305 (G349)		
G306 (G352) G308 (G353) G309 (G358)		
G311 (G359) G313 (G363) G315 (G364)	#1477	ICP, Se, IC %H_2O, OH, TIC, TOC, SPG, GEA, Pu, Am, Np, Cm
G318 (G459) G319 (G460) G323 (G341)	#1476	Sr^{90}, Tc^{99}, C^{14},
Homogeneity Tests (W/Undissolved Solids)		
G301 (G449) G302 (G450) G307 (G453)		
G310 (G454) G317 (G333) G322 (G338)	#1490	GEA (Cs^{137}), ICP (Na), IC (PO_4^{3-})
Composite (W/Undissolved Solids)		ICP, As, Se, Hg, U, NH_4,
G327 (G443)	#1491	IC, %H_2O, OH, TOC, CN, SPG, GEA, Am, Sr^{90}, Tc^{99}, H^3, I^{129}

Table 7. 102-AP Laboratory Sample Identification and Analyte List (continued).

Orig. Sample Shipping No. (Laboratory Aliquot No.)	RSA Number	Analytical Method
IC Comparison (No Solids)		
G299 (G509) G303 (G510) G305 (G511)		
G306 (G521) G308 (G522) G309 (G515)		
G311 (G516) G318 (G517) G319 (G536)	#1574	
G301 (G523) G307 (G527) G317 (G528)		
G322 (G529)	#1575	
G313 (G533) G315 (G534)	#1576	F^-, Cl^-, $NO2^-$ $NO3^-$ SO_4^{2-}, PO_4^{3-}
Full Protocol (No Solids)		
G302 (G477) G310 (G471) 323 (G476)		ICP*, As, Se, Hg, U, NH_4, $\%H_2O$, OH, pH, TIC, TOC, CN, SPG, GEA, Pu, Am, Np, Sr^{90}, Tc^{99}, H^3, C^{14}, I^{129}
G327 (G470)	#1554	

* - ICP Only on G327 (G470)

Hot Cell and Field Blank Samples

Neither a field nor a hot cell blank were required by the TPP for tank 102-AP.

Homogenization Test Samples

In order to prove that the contents of tank 102-AP were homogenous, a total of eighteen samples were analyzed for Cs^{137} (GEA), Na (ICP), and PO_4^{3-} (IC). Twelve of the eighteen samples were ones that were slated for "full" characterization. The other six were special samples that were pulled for the analysis of only the three analytes mentioned in order to obtain enough data to prove homogeneity. The shipping numbers for the six extra "homogenization test" samples were: G301, G302, G307, G310, G317, and G322. The corresponding laboratory aliquot numbers were G449, G450, G453, G454, G333 AND G338 respectively.

Composite Samples for 102-AP

The composite was prepared by mixing equal volumes of each of the individual samples (duplicates not included) and "homogenization' test samples. The proper volume ratios for each sample were determined by a statistical evaluation of the results for Cs, Na, and phosphate (Homogenization Test Samples section).

SECTION II. ANALYSES

Calibrations were performed at the frequencies specified in the analytical procedures and are included with the analysis data for each method. Average results displayed in tables are the average of the sample and duplicate results.

PHYSICAL TEST METHODS

Weight Percent Water (Total Dissolved Solids)

Measurements of true sample water/solid content are obtained with the gravimetric measurements reported in the summaries as weight percent water (wt% water). These measurements were done with approximately 1 ml of sample. Sample aliquots were heated in an oven at 120 °C until replicate measurements demonstrated that the sample was completely dry. Water is lost from simple inorganic salts present in tank farm samples. This procedure applies to the determination of total dissolved solids/percent solids/percent water in solutions, slurries and solid waste. Each are determined in the same manner but are expressed differently. Procedure LA-564-101, Rev. E-3 was used for this analysis. This is a later revision than was called for in the TPP (LA-564-101, Rev. E-1) because the procedure had to be modified to allow for calculating wt% solids rather than just percent water.

All of the individual samples and the composite sample were analyzed in duplicate by this method.

Specific Gravity

The SpG of a substance is defined as its weight per unit volume (density) compared to that of water at 3.98 °C. SpG analysis was done on the full characterization and the composite samples, but not the homogeneity test samples using procedure LA-510-112, Rev. C-2.

PREPARATION METHODS

Acid Digestion

Acid digestions of the samples 102-AP were performed using procedure LA-505-158, Rev. A-2. When a sample plus spike analysis was required the spike was added before the sample digestion, except where there was insufficient sample to do another digestion. For ICP and AA metals, both a post-digestion and a predigestion spike was performed. ICP, As and Se were run on the acid-digested aliquant. Acid digestions were also performed on the standards used to verify instrument calibration and the performance blank. The acid digestions for all samples except G470, G471, G476 and G477 employed a dilution factor of 25. Samples G470-G477 employed a dilution factor of 100. It is believed that (on the samples with a D.F. of 100) a smaller sample size was used in order to conserve sample.

Special Digestion

A special digestion step was required in order to analyze for neptunium, because of the caustic (basic) nature of the tank samples. It was anticipated that this digestion step would also improve the recovery for other actinides, mainly americium. The digestion step consisted of a hydrogen peroxide - nitric acid boil down of the sample, followed by dilution with nitric acid (the standard acid digestion uses HCL, which interferes with actinide extraction). The special digestion steps are part of the approved plutonium/americium method, however it can be applied to neptunium due to its characteristics as an actinide. After some initial results were evaluated, it was determined that the special digestion had essentially no effect on the americium, and was discontinued for the Am analyses. For this reason, some of the Am data employed an additional digestion dilution factor and some didn't. This fact lead to some confusion over which sample data to employ the dilution factor to. Nearly all americium analyses was subsequently performed in order to confirm the initial results. These reruns utilized the special digestion aliquot, and were carefully tracked to ensure that the proper D. F. was performed, documented, and used in the calculations.

Direct/ Routine Dilution

All the remaining analytical methods were performed on either direct sample or analyzed from a dilution of the sample (dilutions are necessary in order to reduce radiation exposure and to remove interferences). These methods were: cyanide, IC, OH, SPG, $\%H^2O$, Hg, ammonia, total organic carbon (TOC), total inorganic carbon (TIC), GEA, ^{14}C, ^{3}H, $^{89/90}Sr$, uranium, ^{99}Tc, and ^{129}I. Only %H2O, SPG and OH were conducted on the direct sample.

Calcium and magnesium contamination often occurs as a result of the powder on the gloves used by laboratory personnel.

Mercury by Cold Vapor Atomic Absorption Spectrophotometry

Mercury analysis was originally completed on the composite only using procedure LA-325-104 Rev. A-0 and Rev. A-1. Additional analyses were then completed on samples G470-G477. These four samples were analyzed in duplicate and were heated prior to analysis to dissolve the solids and keep them in solution. Results for both the composite and the four heated samples were comparable, with all being below detection limits. Due to a different acid digestion dilution factors employed, two different method detection limits were employed. The highest detection limit reported was 0.01 uG/mL.

Inductively Coupled Plasma

Result Reports

The ICP data was reported using a U.S. Environmental Protection Agency Contract Laboratory Package (CLP) software package from WARD Scientific Ltd (WARDS). The data produced by the software, referred to as the WARDS Package, was used to generate the spreadsheet summaries. The WARDs package was modified to report all elements in the WTCP except gadolinium.

WARDS uses the concept of a sample delivery group (SDG), and batches spike, duplicate, instrument control standards, blanks, serial dilutions, interference checks, and narrative information for each sample group. The last character of the SDG (unless this is a rerun analysis) is coded as follows: D for direct, W for water digestion, F for fusion, and A for acid digestion. An R follows this letter for rerun analyses.

In the WARDS package if the result is below the DL, the CLP report format will return a 200% RPD for duplicates and a 100% for serial dilutions. On all pages except the "duplicate" page (form VI-IN), any values less than the DL are replaced with the instrument DL, and should be interpreted as a "less than" value. If the absolute value of the negative result exceeds the DL, the negative value is reported. The duplicate page (form VI-IN) reports what the instrument measures, and may include negative numbers. The units for the "SA" (spike added) column on form V-IN are "uG/L" for both the predigestion and the post digestion spike, however the % Rec. column is the data reported in the batch summary sheet. Post digestion spikes are not listed on the cover page of the WARDs package. Samples G352, G363, G443 and G449 employed a post digestion spike. Sample G443 had a predigestion spike card cut, however no predigestion spike sample was prepared to be analyzed. A predigestion spike was performed on sample G470. If sample concentration before spiking is less than the DL, it is not subtracted for spike calculation.

ICP analyses were performed on acid digestions. In the data summaries for the 102-AP package, the preparation blank and the DL values for digested sample ICP results have been adjusted to account for varying dilution factors and digestion factors between samples, blanks, and detection limits. The detection limits listed in the wards package are instrument detection limits,

whereas the detection limits listed in the summary spreadsheets are method detection limits as they can be compared directly to the results.

The WARDS package is configured to perform interelement corrections for aluminum,iron, magnesium, and chromium.

Quality Control

Failure criteria for an entire WARDs package (batch of samples) are under development. Because each ICP run generates results for 31 elements, there will usually be some failures on each run. These failures are identified in the case narrative associated with each WARDs package. Failure criteria should be based on the absolute failure rate of either spikes, duplicates, LMCS standards, and/or calibration standards, but tempered by the relative importance of the individual element that failed. For example, LMCS and spike recoveries may be generally high for sodium, calcium, and iron from contamination of the blank and sample during preparation, and silicon and barium from the use of glass containers during sample preparation. Spike or LMCS failures noted for these elements should not invalidate the entire WARDS package. LMCS standards that go through the acid digestion often show high results from contamination picked up during preparation, but if the undigested standard recoveries are within limits the sample results in the batch may still be valid.

The case narrative provided with each batch identifies areas that would not meet CLP criteria. All data was evaluated by the QC criteria described on page 9 of this narrative. No direct summaries of this evaluation have been made. Most of the results of the evaluation can be inferred from conclusions reported in the batch narratives. A flag has been added to the data summary sheets to mark those results that were calculated from absorbances that were above the linear calibration range of the instrument. Averages and other calculations and evaluations were sometimes made with this data but are of doubtful use because of the uncertainty in the results.Case (batch) narratives for the data are as follows:

Results

All original ICP analyses had been performed on acid digested aliquots of heated samples. In retrospect, after solids were noticed in the non-digested samples, it was affirmed by laboratory personnel that all heated sample aliquots had been free from solids during the acid digestion ICP preparation procedure. This claim was tested by performing a rerun of sample G433 under the lab Identification of G470, to show reproducibility in the data. The rerun sample was observed closely to confirm that all solids were dissolved prior to and subsequent to digestion/dilution and ICP analysis. A comparison of G470 with the rest of the samples confirmed that all the ICP acid digestions were indeed performed prior to solids formation, and were valid. The mean ICP analyte concentrations were calculated from samples and duplicates.

Uranium

Uranium analysis was performed using procedure LA-925-106, Rev. B-0. The characterization and test plan requires uranium analyssis for the composite only. However, four samples (G470, G471, G476 and G477) were pulled to check whether some uranium may be trapped in the sodium phosphate precipitate which formed when the composite sample was left unheated. Spike is added to all sample aliquots for uranium measurment on the laser florimeter because uranium is quantified by comparing the signal to the known amount. A matrix spike was also added to measure the effect of the matrix on the spike recovery. No end standards were required for the uranium analysis of the samples G470-G477.

Ammonia

Ammonia analysis of 102-AP samples was performed using procedure LA-634-102, Rev. D-0. Ammonia analysis was performed on the composite samples as well as the four solids test samples. All results were either below or near (two times) the detection limit of 1.6E+02 uG/mL. An initial anomaly in the solids test results prompted a rerun of G476 and G477.

Cyanide

Cyanide analysis of 102-AP samples was performed using procedure LA-695-102, Rev. B-0. Cyanide analysis was performed on the composite samples as well as the four solids test samples. Results of all five samples were consistent.

Ion Chromatography

IC analysis of 102-AP samples was performed using procedure LA-533-105, Rev. C-0. The procedure was modified from Rev. B-1, which was specified in the TPP before analysis of any 102-AP samples. IC analysis was performed for the anions fluoride (F), chloride (Cl), nitrite (NO_2), nitrate (NO_3), phosphate (PO_4), and sulfate (SO_4). IC analysis was performed for the composite and individual samples, and on both heated and non-heated (solids present) samples. The heated (rerun) samples were G470, G471, G476, G477, G509, G510, G511, G515, G516, G517, G521, G522, G523, G527, G528, G529, G533, G524 and G536. The results are as follows:

Cl^- Chloride results of the heated verses unheated samples were essentially the same.

NO_2^- Nitrite values of the heated verses unheated samples were essentially the same.

NO_3^- Nitrate values of the heated verses unheated samples were essentially the same.

SO_4^- Sulfate values of the heated verses unheated samples were essentially identical.

PO_4^- Phosphate values of the heated verses unheated samples were significantly different than those taken from supernate of the unheated samples (with solids). Only data from heated samples were used to calculate the concentration of PO4 in the waste.

Total Inorganic Carbon

Total Inorganic Carbon (TIC) analysis was performed using procedure LA-622-102, Rev. B-2. TIC analysis was performed on the composite sample along with the individual samples. Analyses performed on heated aliquots were similar to those taken from non-heated (solids present) samples (G470, G471, G476 and G477).

Total Organic Carbon

Total Organic Carbon (TOC) analysis was performed using procedure LA-344-105, Rev. B-2. TOC analysis was performed on the composite sample along with the individual samples.Analyses performed on heated aliquots were similar to those taken from non-heated (solids present) samples (G470, G471, G476 and G477).

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RADIOANALYTICAL TEST METHODS

Plutonium 239,240

Plutonium-239,240 analysis is performed using procedures LA-503-156, Rev. D-0 and LA-508-051, Rev. A-3. A special acid digestion was performed on some of the samples (See Special Digestion). Plutonium-239,240 analysis was performed on individual samples and on the four solids test samples (G470, G471, G476 and G477), but not on the composite sample (G443). Plutonium-236 tracer is added to the sample and used to correct for chemical recovery, so no extra matrix spike addition is made to the sample. The tracer recovery is listed with the raw data in the chemists batch summary sheet for reference. The sample results for plutonium 239/240 were all below method detection limits. The highest detection limit determined (see detection limit section for radiochemical analyses) was 6.38E-04 μCi/mL. Detection limit for plutonium 239/240 is calculated assuming that the method can not accurately measure a sample that is less than 5% Pu-239/240 (relative to the total percent Pu). The detection limit is therefore based on 0.05 times the total alpha for the sample and corrected for the Pu^{236} recovery.

Plutonium 238

Plutonium 238 is performed using procedures LA-503-156, Rev. D-0 and LA-508-051, Rev. A-3. A special acid digestion was performed on some of the samples (See Special Digestion). All individual samples and the four solids test samples were analyzed. The composite sample (G443) was not analyzed. There is no Pu-238 standard available, therefore no beginning or end standards were analyzed. Plutonium 238 is determined simultaneously with the plutonium-239/240 samples, therefore the results are corrected using the recovery from a plutonium-236 tracer. This tracer is added to the sample and used to correct for chemical recovery, so no extra matrix spike addition is made to the sample. An additional correction is made to account for some Pu^{238} that is contributed by impurities in the Pu^{236} tracer. The tracer recovery is listed with the raw data in the chemists batch summary sheet for reference. The sample results for plutonium 238 were all below method detection limits. The highest detection limit determined (see detection limit section for radiochemical analyses) was 2.2E-04 μCi/mL. Detection limits for plutonium 238 are calculated assuming that the method can not accurately measure a sample that is less than 20 disintegrations per minute (dpm). The detection limit is therefore based on a result of 20 dpm. sample, and corrected for Pu^{236} recovery.

Americium 241

Americium-241 analysis is performed using procedures LA-503-156, Rev. D-0 and LA-508-051, Rev. A-3. A special acid digestion was performed on some of the samples (See Special Digestion). Americium- 241 analysis was performed on the composite as well as the individual samples and the four solid test samples. Americium-243 tracer is added to the sample and used to correct for chemical recovery, so no spike addition is made to the sample. The tracer recovery is listed with the raw data in the chemists batch summary

sheet for reference. The maximum detection limit was determined to be 1.3E-04 μCi/mL. Detection limits for americium 241 are calculated assuming that the method can not accurately measure a sample with an activity that is less than 5% Am-243 peak. The detection limit is therefore based on 0.05 times the Am^{243} contribution.

Strontium-89,90

Strontium-89,90 analyses were performed using procedure LA-220-101, Rev. D-0. Strontium-90 analysis was performed on composite as well as individual samples and the four solids test samples. A $SrCO_3$ carrier is used to correct for chemical recovery, so no spike addition is necessary. The carrier recovery is listed with the raw data in the chemists batch summary sheet for reference. The maximum detection limit was determined to be 1.6E-02 μCi/mL. Detection limits for Sr^{90} are different from sample to sample, and are determined for each measured value (See Detection Limits).

The four solids test samples that were analyzed provided lower Sr^{90} values than did the original samples (0.7 μCi/mL compared to 1.4 μCi/mL). This was unexpected since the rerun samples were heated to dissolve possible Sr-contributing solids, and if anything should have been higher in Sr^{90} concentration. Furthermore, the lowering of apparent strontium concentration seemed to be time dependent (samples that sat the longest period of time prior to analysis had the lowest values). It was hypothesized that the Strontium was plating out on the walls of the sample container or forming some kind of insoluble precipitate over time. A rerun of all the strontium analyses (except the four solids test samples) was performed on the ICP acid digested aliquot that was prepared for each sample to determine which result (0.7 μCi/mL or 1.4 μCi/mL) was correct. The acid digest aliquots were chosen because they represented a dilution of the original samples. A dilute sample has a lower chance of forming solids and/or reacting with the vessel walls. The results of these reruns indicated that the original data was valid.

Technetium-99

Technetium-99 analyses were performed using procedure LA-438-101, Rev. D-1. Technetium-99 analyses were performed on the composite samples as well as the individual samples. Technetium-99 spike was added to measure and correct for chemical recovery of the sample through the separations procedure. In addition, an extra matrix spike was performed.

Normally, only two vials are necessary for a Tc^{99} determination. Samples with the extra matrix spike required four vials (two for each determination) to determine: A) the sample value and B) the sample plus spike value. This technique is not addressed in the Tc^{99} procedure. It is suspected that the four samples were inadvertently placed into the scintillation counter in the wrong order and yielding anomalous results. This theory can be checked out by looking at the actual counting results, arranging them into appropriate order by the magnitude of their relative activities, and recalculating the matrix spikes recoveries.

The maximum detection limit (all samples had unique detection limits based on the background measurement for each sample) was 1.1E-03 μCi/mL.

Carbon-14

Carbon-14 analyses were performed using procedure LA-348-104, Rev. B-0. Carbon-14 analyses were performed on all samples except the composite (G443), and on the four extra solids test samples. A matrix spike was performed on all samples except G459, G470, G476 and G477. Only one of these matrix spikes was required by the TPP. The maximum detection limit was determined to be 4.2E-06 μCi/mL. Detection limits are determined for each measurement, however since the detection limit did not vary (within significant figures) between the sample the duplicate or the blank, a single detection limit value per sample was all that was listed.

Tritium

Tritium analyses were performed using procedure LA-218-114, Rev. A-1 and A-2. This method employs distillation techniques to recover tritiated water vapor from samples. For this reason, tritium must be in the form of tritiated water. Tritium analyses were performed on the composite sample (G443) and the four solids test rerun samples (G470, G471, G476, & G477), but not on the rest of the samples. Matrix spikes were performed on sample G443, however none were required by the TPP. No tritium values could be reported because none of the samples passed all QC criteria. Samples that passed the sample-duplicate RPD failed the matrix spike percent recovery. The procedure mentions that inconsistent results will result if the tritium is in the form of tritiated organic material.

Gamma Energy Analysis

GEA were performed using procedure LA-548-121, Rev. D-0 and LA-508-052, Rev. B-4. GEA were performed on the composite and individual samples, along with the four (heated) solids test samples (G470, G471, G476, lnd G477). Cesium-137 was the only radionuclide found at above detection limits because the ^{137}Cs effectively swamped out the other elements and raised their detection limits. The Cs^{137} limit detection limit employed was the measured D.L. of cesium in the presence of no other significant gamma emitters. This is a valid estimate since no other gamma emitters were detected (all < D. L.). The detection limits for ^{134}Cs, ^{94}Nb, ^{106}RuRh, ^{125}Sb, ^{144}CePr, and ^{60}Co were determined by measuring their respective backgrounds, and applying that value to a statistical equation for determining the level of detection of for radiochemical methods. These individual detection limits are a function of the background contributed by the ^{137}Cs. Since the detection limits vary between sample, duplicate and blank, and also from sample to sample, they will not be listed here, but rather summarized in the summary spreadsheets.

Iodine-129

Iodine-129 analyses were performed using procedure LA-378-103, Rev. B-1. Iodine-129 analyses were performed on the composite and individual samples. The ^{129}I is measured by low-energy gamma counting. Results for ^{129} were at or below detection limits. ^{129}I detection limits are determined for each measurement, however since the detection limit did not vary (within significant figures) between the sample the duplicate or the blank, a single detection limit value per sample group (batch) was all that was listed. The Maximum detection limit listed for the iodine method was 4.8E-05 μCi/mL. No matrix spikes were performed for iodine, since none were required by the TPP.

SECTION III. ACCURACY AND PRECISION STATEMENTS

Accuracy and precision values are listed in table 8. All information for the accuracy and precision statements were taken from data generated between 6-1-92 and 6-1-93. The data does not represent precision for the sample population. Sample precision is addressed in WHC-SD-WM-TRP-168 (Welsh).

Table 6. 222-S Laboratory Measurement Control System Performance Data.

Analyte	EDP Code	Mean	Standard Deviation	3 * Standard Deviation	Lower Control Limit	Upper Control Limit
Ag	S160/S157	100.15	3.73	11.19	88.96	111.34
Al	S102/S101	95.52	4.39	13.17	82.35	108.69
As	S106/S105	98.68	2.85	8.55	90.13	107.23
B	S114/S113	104.03	3.62	10.86	93.17	114.89
Ba	S108/S107	98.00	2.53	7.59	90.41	105.59
Be	S110/S109	100.30	3.68	11.04	89.26	111.34
Bi	S112/S111	102.44	3.24	9.72	92.72	112.16
Ca	S118/S117	97.58	3.75	11.25	86.33	108.83
Cd	S116/S115	98.95	3.49	10.47	88.48	109.42
Ce	S120/S119	104.69	3.12	9.36	95.33	114.05
Cl	R972	102.38	4.23	12.69	89.69	115.07
Cr	S122/S121	101.68	3.01	9.03	92.65	110.71
F	R974	99.20	5.06	15.18	84.02	114.38
Fe	S130/S129	97.73	2.91	8.73	89.00	106.46
K	S152/S149	101.26	3.84	11.52	89.74	112.78
La	S132/S131	100.38	3.16	9.48	90.90	109.86
Li	S136/S135	98.18	2.42	7.26	90.92	105.44
Mg	S138/S137	97.79	3.03	9.09	88.70	106.88
Mn	S140/S139	94.80	2.61	7.83	86.97	102.63
Mo	S144/S141	101.43	3.08	9.24	92.19	110.67
Na	S162/S159	96.81	6.11	18.33	78.48	115.14
Nd	S146/S143	101.15	3.32	9.96	91.19	111.11
Ni	S148/S145	98.51	2.97	8.91	89.60	107.42
P	S150/S147	99.81	4.78	14.34	85.47	114.15
Pb	S134/S133	99.73	3.79	11.37	88.36	111.10
S	S166/S163	99.96	3.82	11.46	88.50	111.42
Sb	S104/S103	99.42	4.98	14.94	84.48	114.36
Se	S156/S153	99.52	4.60	13.80	85.72	113.32
Si	S158/S155	96.81	6.11	18.33	78.48	115.14
Sm	S154/S151	100.58	3.71	11.13	89.45	111.71
Sr	S164/S161	98.24	2.80	8.40	89.84	106.64
Ti	S176/S171	100.05	3.15	9.45	90.60	109.50
Tl	S170/S167	106.44	4.30	18.49	87.95	124.93
Zr	S186/S181	96.86	4.03	12.09	84.77	108.95

Table 6. cont'd 222-S Laboratory Measurement Control System Performance Data.

Analyte	EDP Code elect. data process	Mean	Standard Deviation	3 * Standard Deviation	Lower Control Limit	Upper Control Limit
NO2 ug/ml	R968	101.91	3.92	11.76	90.15	113.67
NO3 ug/ml	R978	101.00	4.55	13.65	87.35	114.65
PO4 ug/ml	R976	101.63	4.61	13.83	87.80	115.46
SO4 ug/ml	R970	103.64	2.83	8.49	95.15	112.13
NH4+ ug/ml	S235	99.78	7.54	22.62	77.16	122.40
Density (g/mL)	S332	98.67	0.62	1.86	96.81	100.53
As ug/ml	R741	104.65	9.43	28.29	76.36	132.94
Hg ug/ml	R716	98.98	7.47	22.41	76.57	121.39
Se ug/ml	R743	100.83	14.53	43.59	57.24	144.42
H2O %wt	S360	100.54	1.24	3.72	96.82	104.26
OH ug/ml	S273	101.00	2.57	7.71	93.29	108.71
CO3/C (TIC) ug/ml	S223	101.10	4.48	13.44	87.66	114.54
TOC (gC/L)	S356	97.30	2.77	8.31	88.99	105.61
CN ug/ml	S244	97.08	2.61	7.83	89.25	104.91
3H uCi/ml	R907	96.79	7.08	21.24	75.55	118.03
60Co uCi/ml	R905	101.90	2.96	8.88	93.02	110.78
137Cs uCi/ml	R901	102.72	2.78	8.34	94.38	111.06
14C uCi/ml	R909	85.12	13.69	41.07	44.05	126.19
99Tc uCi/ml	S363	103.46	10.52	31.56	71.90	135.02
90Sr uCi/ml	S376	100.11	8.07	24.21	75.90	124.32
129I uCi/ml	S298	96.77	17.03	51.09	45.68	147.86
241Am uCi/ml	R201	95.73	11.04	33.12	62.61	128.85
237Np uCi/ml	S380	74.75	12.63	37.89	36.86	112.64
239/240Pu uCi/ml	R211	97.43	7.80	23.40	74.03	120.83

All information was taken from data generated between 6-1-92 and 6-1-93.

SECTION IV. MATERIAL AND CHARGE BALANCES

Material and charge balances calculated with the T-102-AP analysis data were performed by personnel within the process and analytical laboratories. The mass and charge balance calculation results are both very close to "ideal", 99% for charge balance and 101% for mass balance, and lend support to the accuracy of the analytical determinations.

Total μg/mL -Because the samples analyzed were analyzed were liquids, results reported in ug/mL. The total weight per mL of sample was calculated from the following formula.

$$\text{Total } \mu g/mL = 2.2*Al + Cr + Fe + K + Na + Ni + OH^- + 5*TIC + 3*TOC + Cl^- + NO_2^- + NO_3^- + PO_4^{2-} + SO_4^{2-}$$

The aluminum is multiplied by 2.2 because it exists in solution as the aluminate ion AlO_2^-. The ratio of the weight of the ion to the atomic weight of aluminum, 59.0/27.0 equals 2.2. The anhydrous form of aluminate is used rather than the hydrated on, $Al(OH)^-$, because the hydration water is included as part of the $\%H_2O$ analysis. The TIC is multiplied by 5 to convert carbon to carbonate: 60/12 = 5.0.

The TOC is multiplied by 3 to convert carbon to organic compound, including the oxygen, nitrogen, and hydrogen associated with the carbon. The factor of 3.0 is chosen as "typical" for chelator-type organic compounds.

Total μg/g - The volumetric total is converted to a mass-based total by dividing the volumetric (μg/mL) by the specific gravity.

H_2O μg/g - The $\%H_2O$ is multiplied by 10,000 to change the units.

Mass Balance - Sum of "total μg/g" and "H_2O μg/g," divided by 10,000.

Total Cations, eq - The only cations in the solution are Na^+ and K^+, so the total (in micro equivalents per mL) is found by dividing the K concentration by the atomic weight of potassium, dividing the Na value by its atomic weight, and adding the two results together.

Total Anions, eq - The anion total is the result of the following calculation:

$$\text{Total Anions, eq} = Al/27 + OH^-/17 + 2*TIC + Cl^-/35.45 + NO_2^- + NO_3^- /62 + 3*PO_4/95 + 2*SO_4^{3-}/96$$

Aluminum is included in the calculation because it is present in solution as the aluminate ion, as previously discussed. The TIC is multiplied by two because of the -2 charge on the carbonate ion. The divisor is 12 because that is the atomic weight of carbon, and the TIC result is given in μg carbon. The TOC is divided by 36 because there is an average of one negative charge for every three carbon atoms in "typical" chelate-type organic compounds.

Cation/Anion Balance - Total cations divided by total anions.

The material and charge balances obtained are a function of the chemical models used and the selection of the analyte concentrations used in the calculations.

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G335A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G333 102AP-G317
G338 102AP-G322

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in CCV-1 (88.3%).
Potassium in CCV-1 (76.2%) and CCV-4 (81.1%).
Sodium in CCV-1 (85.1%), CCV-2 (238.0%), CCV-3 (78.4%) and CCV-4 (122.5%).

High recoveries for Sodium in CCV-2 can be attributed to memory effects from the samples which contain high levels of Sodium.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Potassium in CCB-1 and CCB-4, Silver in the ICB, Sodium in CCB-1, CCB-2 and CCB-3 and Phosphorus in CCB-1, CCB-2, CCB-3 and CCB-4.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Barium, Iron, Sodium and Phosphorus. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Barium and Iron.

DUPLICATE ANALYSES
Duplicate precision failures were noted as follows:

For G333: Antimony (200.0%), Barium (18.1%) and Iron (19.2%).
For G338: Antimony (200.0%), Barium (18.7%) and Iron (25.5%).

Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. Duplicate failures are attributed to the low level of these analytes in the sample.

S4-7600-076

Hanford Operations and Engineering Contractor for the US Department of Energy

G333 102AP-G317
G338 102AP-G322

LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum and Sodium. These failures may be influenced by the level of contamination found in the preparation blank.

SERIAL DILUTION
A serial dilution failure was noted for Nickel. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. Consequently, serial dilution values were omitted on Form IX.

RECOMMENDATIONS
The values reported for Lead and Potassium should be considered estimates.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G339A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G341 102AP-G323
G342 102AP-G299

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in the ICV (83.6%), CCV-2 (69.7%) and CCV-3 (87.0%).
Potassium in CCV-1 (70.6%), CCV-2 (111.6%) and CCV-3 (123.5%).
Sodium in CCV-1 (76.0%), CCV-2 (202.2%), CCV-3 (267.0%) and CCV-4 (110.3%).
Phosphorus in the ICV (110.9%), CCV-1 (123.3%) and CCV-4 (111.3%).

High recoveries for Sodium in CCV-2 and CCV-3 can be attributed to memory effects from samples containing high levels of Sodium.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Potassium in the ICB, Silver in the ICB and CCB-1, Sodium in the ICB, CCB-1, CCB-2 and CCB-3 and Phosphorus in the ICB, CCB-1, CCB-2 and CCB-4.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Iron, Potassium, Sodium and Phosphorus. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Iron.

DUPLICATE ANALYSES
Duplicate precision failures were noted as follows:

For G341: Antimony (200.0%), Barium (68.4%) and Iron (22.2%).
For G342: Antimony (17.8%), Barium (41.0%) and Iron (19.3%).

Control limits for duplicate samples are based on three times the historical standard deviation of Laboratory Management Control System standards. Duplicate failures are attributed to the low level of these analytes in the sample.

Hanford Operations and Engineering Contractor for the US Department of Energy

G341 102AP-G323
G342 102AP-G299

LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum, Potassium and Sodium. These failures may be influenced by the level of contamination found in the preparation blank.

SERIAL DILUTION
No serial dilution failures were noted. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. Consequently, serial dilution values were omitted on Form IX.

RECOMMENDATIONS
The values reported for Antimony, Lead, Phosphorus and Potassium should be considered estimates.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G346A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G348 102AP-G303
G349 102AP-G305

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in CCV-2 (119.6%), CCV-3 (83.9%) and CCV-4 (115.2%).
Lead in CCV-3 (114.7%) and CCV-4 (111.3%).
Potassium in CCV-2 (129.6%) and CCV-3 (137.0%).
Sodium in CCV-2 (171.3%), CCV-3 (221.6%) and CCV-4 (113.9%).
Phosphorus in the ICV (89.1%) and CCV-3 (110.0%).

High recoveries for Sodium can be attributed to memory effects from the samples which contain high levels of Sodium.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Potassium in CCB-3, Silver in the ICB and CCB-1, Sodium in CCB-2, CCB-3 and CCB-4 and Phosphorus in CCB-1 and CCB-4. The values associated with Potassium and Phosphorus are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Iron, Lead, Sodium and Phosphorus. The values associated with Aluminum, Iron, Lead and Phosphorus are too low to indicate the actual presence of the analyte. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Iron in G359.

DUPLICATE ANALYSES
A duplicate precision failure was noted for Lead (200.0%) in G349. Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. The failure for Lead is attributed to the low level of this analyte in the sample.

G348 102AP-G303
G349 102AP-G305

LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum, Beryllium and Sodium. The low recovery for Beryllium may be attributed to matrix effects of the digestion process.

SERIAL DILUTION
No serial dilution failures were noted. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. A second dilution was analyzed; however, the software is not capable of handling fractional dilution factors. Consequently, serial dilution values were omitted on Form IX. Hand calculation from raw data revealed no serial dilution failures for these elements.

RECOMMENDATIONS
The values reported for Aluminum and Sodium should be considered estimates.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G350A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G352 102AP-G306
G353 102AP-G308

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in the ICV (115.0%) and CCV-4 (123.1%).
Potassium in the ICV (89.1%), CCV-1 (84.6%), CCV-2 (111.3%) and CCV-3 (133.6%).
Sodium in the ICV (89.4%), CCV-1 (74.4%), CCV-2 (168.9%), CCV-3 (207.9%) and CCV-4 (112.1%).
Phosphorus in CCV-1 (75.3%), CCV-2 (70.6%), CCV-3 (71.3%) and CCV-4 (88.1%).

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Silver in the ICB, Sodium in the ICB, CCB-1, CCB-2 and CCB-3 and Phosphorus in ICB, CCB-1, CCB-3 and CCB-4. The values associated with Silver and Phosphorus are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Iron and Sodium. The value associated with Iron is too low to indicate the actual presence of the analyte. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Iron.

POST DIGEST SPIKE SAMPLE RECOVERIES
A spike recovery failure was noted for Potassium (134.1%).

Hanford Operations and Engineering Contractor for the US Department of Energy

G352 102AP-G306
G353 102AP-G308

DUPLICATE ANALYSES

Duplicate precision failures were noted as follows:

For G352: Barium (23.4%), Cadmium (12.4%) and Iron (10.8%).

For G353: Barium (18.4%), Beryllium (15.9%) and Iron (9.1%).

Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. Failures for Barium, Beryllium and Cadmium are attributed to the low level of these analytes in the sample. Failures for Iron may be influenced by the values reported for the preparation blank.

LABORATORY CONTROL SAMPLE

Failures were noted for Aluminum, Antimony, Iron, Silver and Sodium. Failures for Sodium may be influenced by the level of contamination found in the preparation blank. The low recovery for Silver may be attributed to matrix effects of the digestion process.

SERIAL DILUTION

No serial dilution failures were noted. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. A second dilution was analyzed for G352; however, the software is not capable of handling fractional dilution factors. Consequently, serial dilution values were omitted on Form IX. Hand calculation from raw data revealed no serial dilution failures for Aluminum, Sodium or Phosphorus. No serial dilution evaluation is possible for Sodium in G353.

RECOMMENDATIONS

The values reported for Aluminum, Antimony, Iron, Potassium, Silver, Sodium and Phosphorus should be considered estimates.

ABBREVIATIONS

* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G356A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G358 102AP-G309
G359 102AP-G311

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in CCV-1 (83.7%), CCV-2 (113.8%), CCV-3 (73.7%) and CCV-4 (124.5%).
Potassium in CCV-1 (111.3%), CCV-2 (156.0%) and CCV-3 (164.9%).
Sodium in CCV-2 (196.0%), CCV-3 (227.1%) and CCV-4 (118.9%).
Phosphorus in CCV-2 (112.2%) and CCV-3 (119.6%).

High recoveries for Sodium can be attributed to memory effects from the samples which contain high levels of Sodium.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Aluminum in CCB-1, CCB-2, CCB-3 and CCB-4, Chromium in CCB-2, Lead in the ICB and CCB-3, Nickel in CCB-3, Potassium in CCB-2 and CCB-3, Silver in the ICB, CCB-1 and CCB-2, Sodium in CCB-2, CCB-3 and CCB-4, and Phosphorus in CCB-1 and CCB-4. The values associated with Aluminum, Chromium, Lead, Nickel and Phosphorus are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Iron, Lead, Nickel, Potassium, Sodium and Phosphorus. The values associated with Lead, Nickel, Potassium and Phosphorus are too low to indicate the actual presence of the analyte.

84 7600-076

Hanford Operations and Engineering Contractor for the US Department of Energy

G358 102AP-G309
G359 102AP-G311

DUPLICATE ANALYSES
Duplicate precision failures were noted as follows:
For G358: Cadmium (12.2%) and Iron (122.7%).
For G359: Antimony (200.0%), Iron (14.1%) and Lead (200.0%).
Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. These failures are attributed to the low level of these analytes in the sample. The failure for Iron in G358 may be influenced by the value reported for the preparation blank.

LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum and Sodium.

SERIAL DILUTION
A serial dilution failure was noted for Nickel. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. A second dilution was analyzed; however, the software is not capable of handling fractional dilution factors. Consequently, serial dilution values were omitted on Form IX. Hand calculation from raw data revealed no serial dilution failures for Aluminum, Sodium and Phosphorus.

RECOMMENDATIONS
The values reported for Aluminum, Nickel and Sodium should be considered estimates.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G361A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G363 102AP-G313
G364 102AP-G315

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Potassium in CCV-1 (80.5%), CCV-2 (112.2%) and CCV-4 (87.8%).
Sodium in the ICV (47.0%), CCV-1 (12.4%), CCV-3 (127.0%) and CCV-4 (14.2%).
Phosphorus in CCV-1 (82.0%), CCV-3 (70.7%) and CCV-4 (76.6%).

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Potassium in CCB-1, Silver in the ICB, Sodium in all calibration blanks and Phosphorus in CCB-1, CCB-2, CCB-3 and CCB-4. The values associated with Potassium and Phosphorus are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Cadmium, Iron, Lead and Sodium. The values associated with Aluminum, Cadmium, Iron and Lead are too low to indicate the actual presence of the analyte. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Iron.

POST DIGEST SPIKE SAMPLE RECOVERIES
A spike recovery failure was noted for Silver (10.0%). The low recovery for Silver may be attributed to matrix effects of the digestion process.

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Hanford Operations and Engineering Contractor for the US Department of Energy

G363 102AP-G313
G364 102AP-G315

DUPLICATE ANALYSES

Duplicate precision failures were noted as follows:

For G363: Antimony (52.5%) and Iron (15.3%).

For G364: Barium (61.1%), Beryllium (18.0%), Cadmium (11.2%), Iron (13.9%) and Lead (200.0%).

Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. Failures are attributed to the low level of these analytes in the sample.

LABORATORY CONTROL SAMPLE

Failures were noted for Aluminum, Lead, Potassium and Silver. Low recoveries for Lead and Silver may be attributed to matrix effects of the digestion process. The low recovery for Potassium is consistent with the low bias found in the calibration verification standards. The high recovery for Aluminum cannot be readily explained by contamination found in the preparation blank; however, the recovery of undigested standards were within specifications.

SERIAL DILUTION

No serial dilution failures were noted. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. A second dilution was analyzed; however, the software is not capable of handling fractional dilution factors. Consequently, serial dilution values were omitted on Form IX. Hand calculation from raw data revealed no serial dilution failures for Aluminum, Sodium and Phosphorus.

RECOMMENDATIONS

The values reported for Potassium and Phosphorus should be considered estimates. Calibration verification standards (500 ppb) and blanks indicate that Sodium is unusable; however, undigested control standards (10 ppm) were in specification. The samples were measured at ca. 80 ppm. Without a high standard to bracket the sample concentration, Sodium values are suspect.

ABBREVIATIONS

* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G441A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G443 102AP-G327

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Potassium in CCV-1 (83.2%), CCV-2 (117.5%) and CCV-3 (81.2%).
Sodium in the ICV (76.3%), CCV-1 (12.4%), CCV-2 (126.9%) and CCV-3 (15.4%).

High recoveries for Sodium and Potassium in CCV-2 can be attributed to memory effects from the samples which contain high levels of these analytes. The low bias in the other CCV's is compensated by adequate recoveries (>95%) from 10 ppm standards analyzed before and after the analytical run.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Silver in the ICB and CCB-1 and Sodium in all calibration blanks.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Cadmium, Iron, Lead and Sodium. The values associated with Cadmium and Lead are too low to indicate the actual presence of the analyte. Considering the value reported for the preparation blank and the value reported for the sample, a false positive may exist for Iron.

POST DIGEST SPIKE SAMPLE RECOVERIES
No spike recovery failures were noted. A post digestion spike was analyzed in lieu of a matrix spike which was not prepared.

Hanford Operations and Engineering Contractor for the US Department of Energy

G443 102AP-G327

DUPLICATE ANALYSES

Duplicate precision failures were noted for Barium (55.6%) and Iron (29.5%). Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. The failures for Barium is attributed to the low level of this analyte in the sample. The failure for Iron may be influenced by the value reported for the preparation blank.

LABORATORY CONTROL SAMPLE

Failures were noted for Aluminum, Iron, Silver and Sodium. Control limits are based on three times the historical standard deviation of Labratory Management Control System standards. The failure for Sodium may be influenced by the value reported for the preparation blank. The low recovery for Silver may be attributed to matrix effects of the digestion process.

SERIAL DILUTION

Serial dilution failures were noted for Nickel. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, a 51-fold dilution was analyzed and the values were reported on Form I after appropriate dilution factors were applied. Consequently, serial dilution values were omitted on Form IX.

RECOMMENDATIONS

The values reported for Nickel, Iron and Aluminum should be considered estimates.

ABBREVIATIONS

* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G447AR
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G449 102AP-G301
G450 102AP-G302

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in CCV-1 (117.1%), CCV-2 (150.8%) and CCV-4 (123.3%).
Potassium in CCV-1 (84.6%), CCV-2 (133.7%) and CCV-3 (148.3%).
Sodium in the ICV (85.1%), CCV-1 (55.6%), CCV-2 (194.0%) and CCV-3 (241.3%).
Phosphorus in CCV-1 (79.2%).

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Silver in the ICB, Sodium in all calibration blanks and Phosphorus in CCB-1 and CCB-3. The values associated with Phosphorus are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Iron and Sodium. The value associated with Sodium is too low to indicate the actual presence of the analyte. Considering the values reported for the preparation blank and the values reported for the sample, a false positive may exist for Iron.

POST DIGEST SPIKE SAMPLE RECOVERIES
No spike recovery failures were noted.

DUPLICATE ANALYSES
Duplicate precision failures were noted as follows:

For G449: Beryllium (28.1%) and Iron (33.5%).
For G450: Antimony (200.0%) and Cadmium (14.8%).

Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. Failures are attributed to the low level of these analytes in the sample.

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Hanford Operations and Engineering Contractor for the US Department of Energy

G449 102AP-G301
G450 102AP-G302

LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum, Beryllium, Iron, Sodium and Phosphorus. Failures for Aluminum may be influenced by the level of contamination found in the preparation blank. Low recoveries for Beryllium and Phosphorus may be attributed to matrix effects of the digestion process.

SERIAL DILUTION
Serial dilution failures were noted for Nickel. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. A second dilution was analyzed for G449; however, the software is not capable of handling fractional dilution factors. Consequently, serial dilution values were omitted on Form IX. Hand calculation from raw data revealed no serial dilution failures for Aluminum, Sodium and Phosphorus. No serial dilution evaluation is possible for Sodium in G450.

RECOMMENDATIONS
These data represent a re-analysis of the original sample preparation. The values reported for Antimony, Beryllium, Iron, Nickel, Potassium, Sodium and Phosphorus should be considered estimates.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G451A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G453 102AP-G307
G454 102AP-G310

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in CCV-2 (87.7%), CCV-3 (88.1%) and CCV-4 (86.3%).
Lead in CCV-4 (89.7%).
Potassium in CCV-1 (83.7%), CCV-2 (111.0%), CCV-3 (133.6%) and CCV-4 (89.3%).
Sodium in CCV-1 (67.9%), CCV-2 (185.6%) and CCV-3 (248.8%).
Phosphorus in the ICV (111.1%), CCV-1 (120.2%), CCV-2 (118.0%), CCV-3 (125.1%) and CCV-4 (121.5%).

High recoveries for Sodium in CCV-2 and CCV-3 can be attributed to memory effects from samples containing high levels of Sodium.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Silver in the ICB and CCB-1, Sodium in the ICB, CCB-1, CCB-2 and CCB-3 and Phosphorus in all calibration blanks.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Cadmium, Iron, Lead, Sodium and Phosphorus. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Iron.

DUPLICATE ANALYSES
Duplicate precision failures were noted as follows:

For G453: Antimony (200.0%), Barium (45.6%), Cadmium (16.9%) and Iron (42.6%).
For G454: Cadmium (22.9%) and Lead (200.0%).

Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. Duplicate failures for Antimony, Barium, Cadmium and Lead are attributed to the low level of these analytes in the sample.

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G453 102AP-G307
G454 102AP-G310

LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum, Potassium and Sodium.

SERIAL DILUTION
Serial dilution failures were noted for Chromium and Nickel.

RECOMMENDATIONS
The values reported for Antimony, Iron, Lead, Potassium and Phosphorus should be considered estimates.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G457A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G459 102AP-G318
G460 102AP-G319

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Antimony in CCV-3 (89.9%) and CCV-4 (71.4%).
Potassium in CCV-1 (72.5%), CCV-2 (89.9%) and CCV-3 (132.0%).
Sodium in CCV-1 (73.5%), CCV-2 (199.4%), CCV-3 (246.2%) and CCV-4 (88.0%).
Phosphorus in the ICV (113.1%), CCV-1 (118.0%), CCV-2 (113.1%), CCV-3 (112.2%) and CCV-4 (116.6%).

High recoveries for Sodium can be attributed to memory effects from the samples which contain high levels of Sodium.

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Potassium and Silver in the ICB, Sodium in the ICB, CCB-1, CCB-2 and CCB-3, and Phosphorus in CCB-1, CCB-2 and CCB-4. ~~The~~ values associated with Potassium and Phosphorus are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Iron, Sodium and Phosphorus. The values associated with Phosphorus are too low to indicate the actual presence of the analyte. Considering the values reported for the preparation blank and the values reported for the sample, false positives may exist for Iron.

64 7600-076

Hanford Operations and Engineering Contractor for the US Department of Energy

G459 102AP-G318
G460 102AP-G319

DUPLICATE ANALYSES

Duplicate precision failures were noted as follows:

For G459: Barium (8.9%), Beryllium (14.9%) and Iron (13.1%).

For G460: Barium (19.6%) and Iron (9.4%).

Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. Failures for Barium and Beryllium and Iron in G459 are attributed to the low level of these analytes in the sample. The failure for Iron in G460 may be influenced by the value reported for the preparation blank.

LABORATORY CONTROL SAMPLE

Failures were noted for Aluminum, Barium, Cadmium, Chromium, Potassium, Silver and Sodium. Control limits are based on three times the historical standard deviation of Labratory Management Control System standards. Failures for Aluminum and Sodium may be influenced by the values reported for the preparation blank.

SERIAL DILUTION

Serial dilution failures were noted for Nickel. The original sample values for Aluminum, Sodium and Phosphorus exceeded the linear range of the instrument. As a result, the serial dilution values were reported on Form I after appropriate dilution factors were applied. A second dilution was analyzed; however, the software is not capable of handling fractional dilution factors. Consequently, serial dilution values were omitted on Form IX. Hand calculation from raw data revealed no serial dilution failures for these elements.

RECOMMENDATIONS

The values reported for Barium, Cadmium, Chromium, Nickel, Potassium and Silver should be considered estimates.

ABBREVIATIONS

* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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G468A
CASE NARRATIVE - ICP

WHCPAL LABORATORIES
WHCPAL/CUSTOMER SAMPLE NOS: G470 102AP-G327

INORGANIC ANALYSIS

The data evaluation for this project will follow the guidelines specified in "Technical Project Plan for the 222-S Laboratory in Support of the Grout Treatment Facility Sampling and Characterization Plans for Tanks 105-AP, 106-AP and 102-AP", WHC-SD-WM-TPP-008. Contained in this report is the evaluation of the data generated by Westinghouse Hanford Company - Processing and Analytical Laboratories (WHC-PAL), 222-S Laboratory. Information will be organized by quality control parameter. No computer-readable data will be submitted on floppy diskette as mentioned in the comment on the following cover page.

INITIAL AND CONTINUING CALIBRATION VERIFICATION
All instrument calibration requirements were in specification during analysis, with the exceptions noted below:

Aluminum in CCV-1 (118.9%) and CCV-3 (112.7%).
Antimony in CCV-3 (66.4%).
Potassium in CCV-3 (79.9%).
Phosphorus in CCV-1 (113.1%).

INITIAL AND CONTINUING CALIBRATION BLANKS
All ICB and CCB analytical measurements were within the IDL with the exception of Silver in the ICB and CCB-1 and Phosphorus in CCB-1. The values associated with these exceptions are too low to indicate the actual presence of the analyte.

PREPARATION BLANK
All analytical measurements were within the IDL with the exception of Aluminum, Antimony, Iron and Sodium. The values associated with these exceptions are too low to indicate the actual presence of the analyte.

SPIKE SAMPLE RECOVERIES
A spike recovery failure was noted for Silver (26.3%). A matrix effect of the digestion process is suspected in the failure for Silver.

DUPLICATE ANALYSES
A Duplicate precision failure was noted for Nickel (21.6%). Control limits for duplicate samples are based on three times the historical standard deviation of Labratory Management Control System standards. The failure for Nickel is attributed to the low level of this analyte in the sample.

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LABORATORY CONTROL SAMPLE
Failures were noted for Aluminum, Iron, Silver and Sodium. The failure for Iron may be influenced by the value reported for the preparation blank. The low recovery for Silver may be attributed to matrix effects of the digestion process.

SERIAL DILUTION
No serial dilution failures were noted.

RECOMMENDATIONS
The values reported for Aluminum, Iron, Nickel and Sodium should be considered estimates. Values reported for Silver are unusable.

ABBREVIATIONS
* = Duplicate failure. E = Serial dilution failure. N = Spike failure. NR = Not required. P = ICP method used. U = less than IDL.

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