START

0018207

Final
May 29, 1991

Meeting Minutes Transmittal/Approval
Unit Managers Meeting: Grout Treatment Facility

Meeting Held March 22, 1991
Westinghouse Hanford Lacey Office
Lacey, Washington

Appvl. _____ Date: 5/30/91
Clifford E. Clark, Environmental Policy and Permitting, DOE-RL

Appvl. _____ Date: 5/30/91
Daniel L. Duncan, EPA Region 10, RCRA Program Manager

Appvl. _____ Date: 5/30/91
~~Jeff Voogd~~, Unit Manager, Contractor Representative, WHC
Ralph Wood

Appvl. _____ Date: 5-30-91
Susan M. Price, RCRA Permitting, Contractor Representative, WHC

Appvl. _____ Date: 5/30/91
Joseph Witczak, Unit Manager, Washington State Department of Ecology

PURPOSE:
Meeting Minutes are attached. These minutes are from the March Unit Managers Meeting held 3-22-91. Minutes are comprised of the following:

Attachment 1 - Summary of Discussion and Commitments
Attachment 2 - Attendance List
Attachment 3 - Agenda
Attachment 4 - Action Items List with Status
Attachment 5 - Irradiation Testing of an Asphalt-based Grout Vault Sealant
Attachment 6 - Gas Generation and Release From Double-Shell Slurry Feed Grout Vaults
Attachment 7 - Tank 241-AN-106 Characterization Results
Attachment 8 - Action Item 1-29-91:1 (information of PQLs)
Attachment 9 - Monthly Report, March

JAN 1992
RECEIVED
EDMC

9 2 1 2 4 1 4 7 3 9 1

Attachment #1

Grout Treatment Facility
Unit Managers Meeting

Washington State Department of Ecology Office
Kennewick, Washington
March 22, 1991

Summary of Discussion and Commitments

1. Tom Woebkenberg (SWEC) was introduced. He will be the program contact in Waste Management for Grout, and will be functioning as a DOE person. He is also the site rep.

2. Joe Westsik (PNL) gave a presentation on the radiation test results on the asphalt liner. The handout from this presentation is included as Attachment #5. A 300-year maximum dose was used, which is an order of magnitude greater than the 30-year monitoring period. The testing found changes in the properties of the liner materials, but none of the changes showed deficiencies that would eliminate using the NOKORODE material. The report is in final review.

ACTION ITEM: The final report will be given to Joe Witczak (Ecology) and Dan Duncan (EPA) as soon as it is available. Action: WHC

3. Jeff Voogd (WHC) handed out to the regulators the hydrogen gas generation and release report. A copy of the report is included as Attachment #6. The report is the basis for design changes, which have already been incorporated. 14 to 18 of the vaults will be double-shell slurry feed. The first vault is a mix of phosphate sulfate wastes.

4. Copies of the characterization report for the 106-AN tank were handed out to the regulators. This report is included as Attachment #7. This is an example of the sampling process to be used for the candidate tanks. For the actual feed tank, it is expected to have a RCRA compliant characterization package, showing all the agreed to protocols have been observed. A discussion was held on the land disposal restrictions (LDR). Cliff Clark (DOE-RL) stated that they considered the point of application of the LDR to be the candidate tank. He further stated that EPA could tell DOE-RL that the evaporator treatment is sufficient to meet BDAT, and then the material could be placed in the vault. No decision was reached.

5. A discussion was held on permit modifications. Joe Witczak (Ecology) stated that the new state regulations will be effective April 7th, and that they contain instructions for permit modifications. Cliff Clark (DOE-RL) stated that they will recommend annual submittals for all Class I modifications at one time.

6. A discussion was held of the difference between practical quantitation limits (PQLs) and detection limits. Jeff Voogd provided a handout

(Attachment #8), which was discussed. This was in response to Action Item 1:29-91:1. Cliff Clark (DOE-RL) stated that he would like to see the permit reflect the requirements which the lab would have to meet, not what can be accomplished in the process.

7. A discussion of NOD comments/responses was held.

o NODs 6 and 8 will be resolved in the Sitewide meetings, and the text will be revised in accordance with those decisions.

o In response to NOD 9, the application will be modified to include bottle-on-a string and non-destructive testing, as well as core drilling.

o With regard to NOD 17, Joe Witczak (Ecology) stated that he was interested in using the list of organic constituents given in WAC 173-303-9903, but that he was not implying, by using that particular list, that the wastes were either U- or P-listed wastes. Cliff Clark (DOE-RL) asked for clarification on whether sampling would have to be conducted for the entire list. Joe Witczak stated that it was an "interest list", and agreed that while WHC would not necessarily analyze for all of the parameters on 9903, should they detect, through whatever analysis, any constituent listed on 9903, they would inform Ecology.

o The gas generation information presented was in response to NOD 20.

o In response to NOD 25, Stan Hill (WHC) stated that they would comply with EPA Guidance Document 530-SW-86-016, in the event that free liquids were found in the landfill.

o In response to NOD 26, WHC is going to revise the application to state that core drilling will be performed until there is increased confidence in the other testing techniques.

o For NOD 27, bottle-on-a-string samples will be taken from the first DST vault until Ecology is fairly confident in the nondestructive techniques.

o For NOD 29, pretreatment design and formulation options are currently being evaluated. Ecology is waiting for the report to review, but the report is still in clearance.

o For NOD 36, the text is revised to state that if a sludge layer is found, sampling will be done.

o NOD 38 has been deferred to Sitewide, or beyond..

o NODs 53 and 54 will be closed.

o For NOD 56, the text will be revised, as requested.

o For NOD 59, the tank integrity assessment plan has been drafted, and it is anticipated that it will be in the July permit.

o For NOD 68, a report will be submitted, comparing the actual chemical composition of the leachate sump liquids to the simulants used in the compatibility test. The report will be submitted to Ecology within 90 days after the grout has solidified and the grout temperature is no longer increasing.

o For NOD 124, Stan Hill (WHC) stated that while earlier they had agreed to add the requirement to the permit, but now they are backing off. The issue should be handled through the Tri-Party Agreement and not the permit, due to the permit modification process.

o For NODs 142 and 143, like 29, are waiting for the report on adiabatic calorimetry. A discussion was held on the question of the level of technical detail which should be included in the permit. Cliff Clark (DOE-RL) stated that he felt that there shouldn't be any data in the permit application, but rather it should be in the supporting documentation, to support some of the concepts. Gary Anderson (Ecology) will make a recommendation to Joe Witczak (Ecology) on a proposal for rewording the NOD comments.

o For NOD 203, WHC is going to incorporate the response.

o For NOD 207, the text has been modified.

o For NOD 227, DOE and WHC will discuss the proposed response.

o For NOD 228, the wording will be modified to reflect the aisle space required in the new Ecology regulations.

o For NOD 233, the Sitewide meetings will handle spill reporting issues.

o For NOD 246, Stan Hill (WHC) stated that initially the vault will be a surface impoundment, with free liquid on top of the grouted waste until the excess water is removed. WHC is suggesting that while the grout is a surface impoundment a 20 gallon per day leak rate be used, and then lower it from 20 down to 5 gallons per day subsequent to removal of free water.

o For NOD 247, it was stated that at postclosure, WHC will submit data in order to obtain approval to reduce the reporting period.

o For NOD 250, the detailed grout sampling plan will be completed in FY 91. The first sample will be extensively analyzed and future testing will be less extensive unless significant differences are found. The minimum volume accumulated or time expired between sampling will be addressed in the sampling plan.

o For NOD 251 the RAP has been revised.

o For NOD 263, Ecology's comments have been incorporated into the table.

o With respect to NOD 264 the blending ratios are generally 50%. Blending ratios for other waste types (NCAW, NCRW, etc.) are to be determined at a later time.

o For NOD 269 the information has been provided in Appendix 3J.

o For NOD 273 the report is going through clearance.

o For NOD 288 the reference was provided at the January UMM.

o For NOD 295 the permit application will be revised to remove the wording that excess water will be returned.

o For NOD 297 a statement on chemical compatibility has been added as requested.

o NODs 298 and 301 will be resolved in the Sitewide meetings.

o For NOD 302 the CQA plan has been updated to state that draft NCRs will go directly to Ecology.

o For NOD 324 the model has been updated to included the actual physical conditions.

o For NOD 289 the response is that this is a disposal facility and that recovery and recycling is not the subject here.

8. The schedule was discussed. The certified permit submission date is moved from June to July/August, assuming all the NODs can be closed. Another round of NOD comments could put the schedule off by six months.

The next meeting will take place on April 29, 1991 in the afternoon, in Richland, Washington.

Attachment 2

Grout Treatment Facility
Unit Managers Meeting
March 22, 1991

Attendance List

NAME	ORGANIZATION	PHONE #
G. Anderson	Ecology	(206) 438-7558
C. Clark	DOE-RL	(509) 376-9333
D. Duncan	EPA	(206) 553-6693
K. Davis	SWEC	(509) 376-0412
S. Hill	WHC	(509) 376-1674
J. King	SWEC	(509) 376-4726
S. Price	WHC	(509) 376-1653
J. Voogd	WHC	(509) 373-5642
J. Westsik	PNL	(509) 376-5985
J. Witczak	Ecology	(206) 438-7557
T. Woebkenberg	SWEC	(509) 376-1177
R. Wood	WHC	(509) 373-4731

Attachment 4

Grout Treatment Facility Unit Managers Meeting

Commitments/Agreements Status (Status date: 3/22/91)

ACTION ITEM	COMMITMENTS/AGREEMENTS STATUS LIST
7-23-90:3	A copy of the TPA change request will be sent to Dan Duncan with the EPA. Action: Cliff Clark.
	OPEN
9-18-90:3	WHC will provide updated Bluelines at the January 1991 UMM. Action: Steve Briggs.
	OPEN This item is 80% complete.
1-4-91:1	WHC to provide information on the rate of hydrogen build up within the vault and the rate of diffusion of hydrogen out of the vault. Action: Jeff Voogd.
	CLOSED
1-29-91:1	WHC and DOE will provide greater detail on the practical quantitation limits and the relationship with the contract laboratory procedures by the next Unit Managers Meeting. Action: Jeff Voogd.
	CLOSED - information provided at 3-22-91 meeting.
1-29-91:2	EPA Region X will inquire of EPA-HQ where in the waste stream the designation and regulations apply. Action: Dan Duncan.
	OPEN
1-21-91:3	WHC will prepare a letter for DOE-RL through Elizabeth A. Bracken to EPA Region X addressing the following items: 1) a statement of the nature of the wastes coming out of the candidate tanks; 2) a statement that in treating the effluent an apparent nonwaste water is created, whereas heretofore the tank effluent may have been a non-waste water; 3) Does the reduced concentration of TOC in the treated waters mandate their designation as waste water? Action: Jeff Voogd.
	CLOSED SW 5-30-91

3-22-91:1 The final report on the radiation test results will be given to Joe Witczak (Ecology) and Dan Duncan (EPA) as soon as it is available. Action: WHC

OPEN

9 1 1 2 4 1 1 0 0 9 9

#5

IRRADIATION TESTING OF AN ASPHALT-BASED GROUT VAULT SEALANT

GROUT UNIT MANAGERS MEETING

MARCH 22, 1991

GROUT VAULT LINER IRRADIATION STUDIES

A. NOKORODE 705M WAS GAMMA-IRRADIATED TO MAX. DOSE EXPECTED FOR 300-YEAR LIFETIME, IN CONTACT WITH SYNTHETIC DSSF AND PORE FLUID.

B. MEASUREMENTS WERE MADE TO DETECT CHANGES IN DENSITY, WATER-TIGHTNESS, ADHESION TO CONCRETE, TENSILE PROPERTIES AND PENETRATION RESISTANCE.

C. THE CHEMICAL AND RADIATION ENVIRONMENTS CAUSED MEASURABLE CHANGES IN THE NOKORODE, BUT

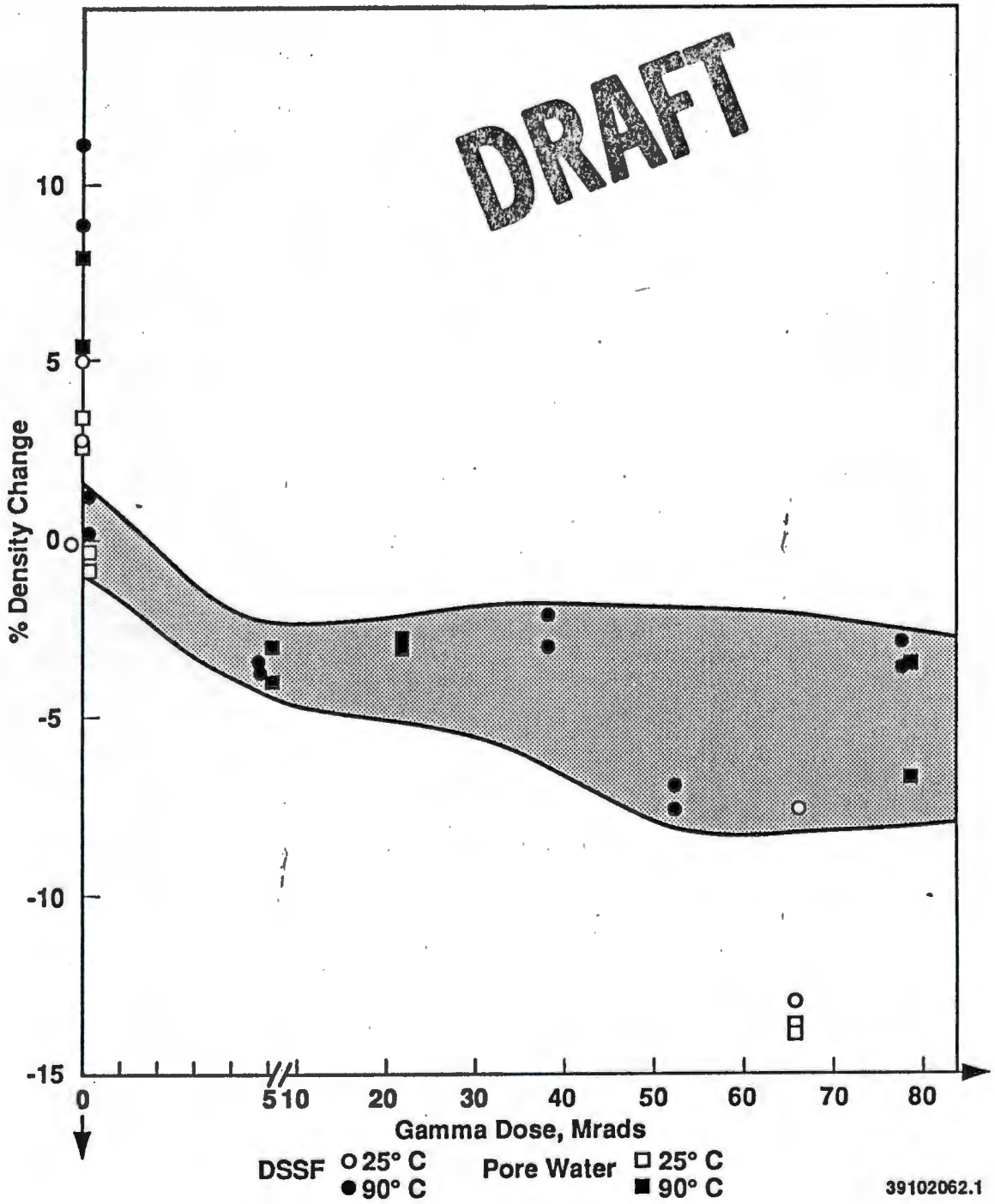
D. NONE OF THE TESTING SHOWED DEFICIENCIES WHICH WOULD ELIMINATE NOKORODE FROM FURTHER CONSIDERATION.

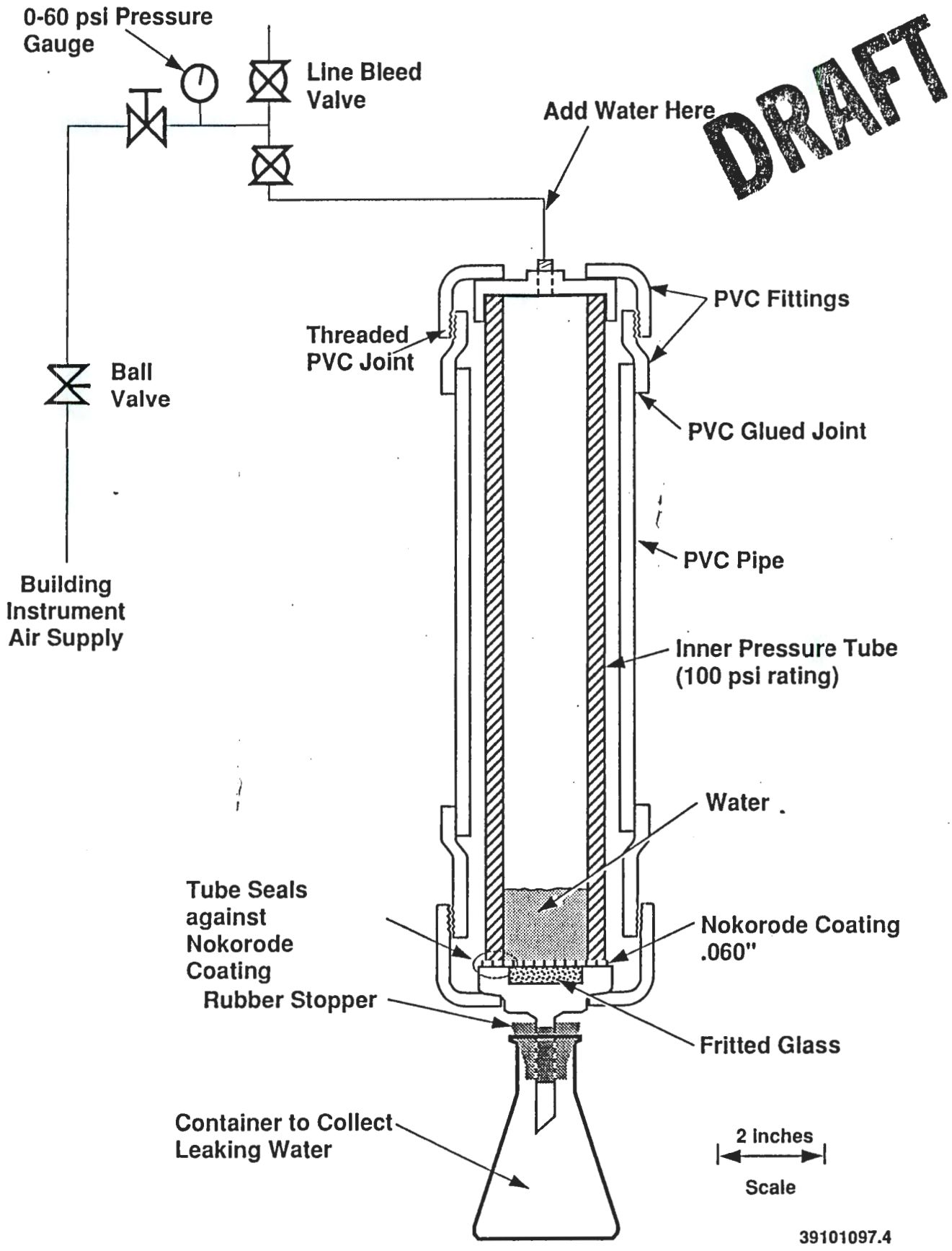
DRAFT

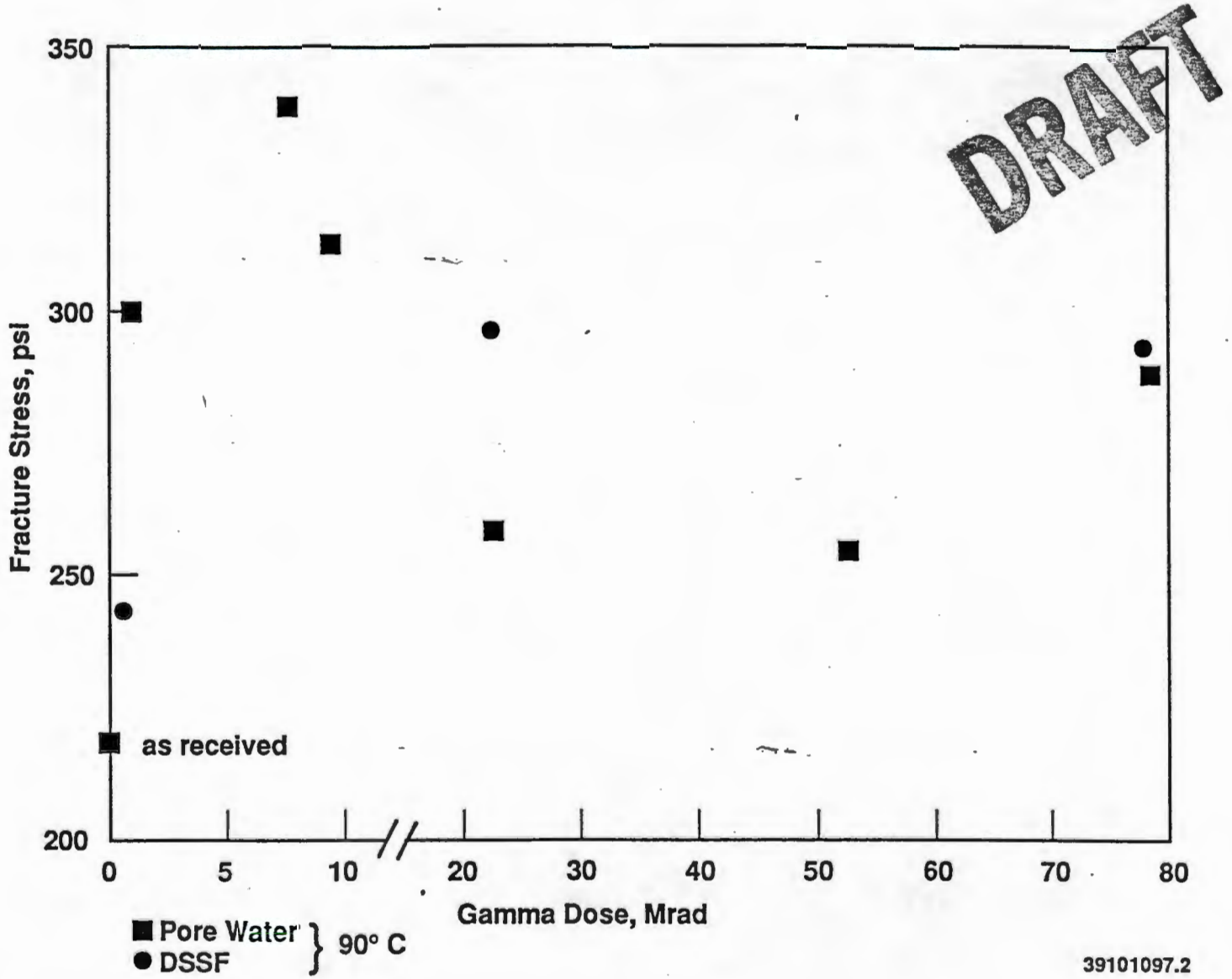
DRAFT

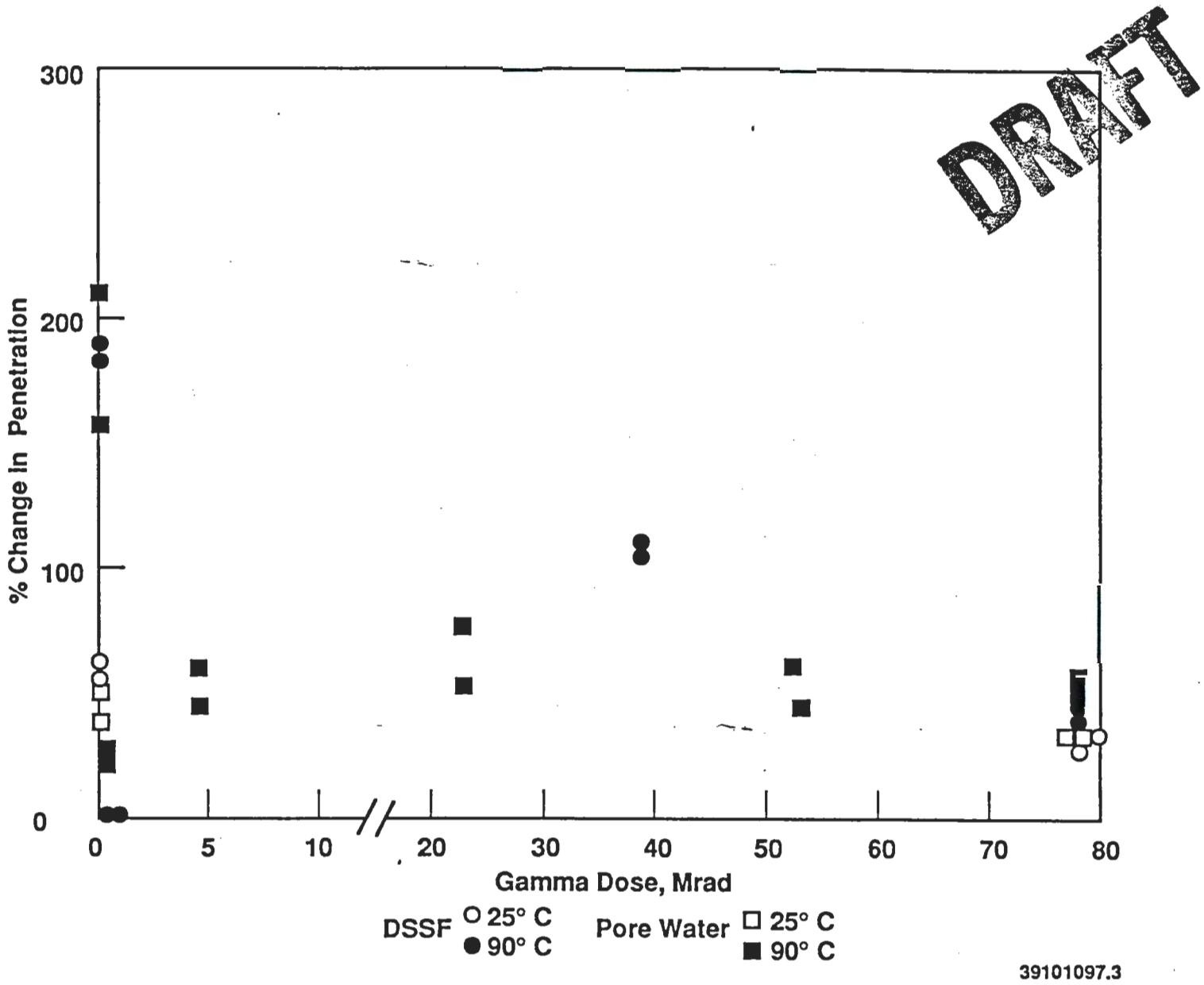
TABLE 1. Exposure Conditions of Specimens

Sample Type	Environment	Temperature, °C	Dose, MRad	Total Samples
Cup	DSSF	25	0, 78	4
Cup	Pore Water	25	0, 78	4
Cup	DSSF	90	0, 0.152, 8.54, 38.9, 78	10
Cup	Pore Water	90	0, 0.152, 8.54, 38.9, 78	10
Fritted Filter	DSSF	90	0, 38.9	4
Fritted Filter	Pore Water	90	0, 38.9	4
Coated Concrete	DSSF	90	0, 0.152, 8.54, 38.9, 78	10
Coated Concrete	Pore Water	90	0, 0.152, 8.54, 38.9, 78	10
Dogbone	DSSF	90	0, 0.152, 8.54, 38.9, 78	10
Dogbone	Pore Water	90	0, 0.152, 8.54, 38.9, 78	10


DRAFT
% Density Change
10
5
0
-5
-10
-15
0
5 10
20
30
40
50
60
70
80
Gamma Dose, Mrads
DSSF
25° C
90° C
Pore Water
25° C
90° C
39102062.1


0-60 psi Pressure Gauge
Line Bleed Valve
Add Water Here
DRAFT
PVC Fittings
Threaded PVC Joint
Ball Valve
PVC Glued Joint
PVC Pipe
Building Instrument Air Supply
Inner Pressure Tube (100 psi rating)
Water
Tube Seals against Nokorode Coating
Nokorode Coating .060"
Rubber Stopper
Fritted Glass
Container to Collect Leaking Water
2 Inches
Scale
39101097.4


DRAFT
350
300
250
200
Fracture Stress, psi
as received
0
5
10
20
30
40
50
60
70
80
Gamma Dose, Mrad
Pore Water
DSSF
90° C
39101097.2


DRAFT
300
200
100
0
% Change In Penetration
0
5
10
20
30
40
50
60
70
80
Gamma Dose, Mrad
DSSF
25° C
90° C
Pore Water
25° C
90° C
39101097.3

#6

PNL-7644

HANFORD GROUT TECHNOLOGY PROGRAM

GAS GENERATION AND RELEASE FROM DOUBLE-SHELL SLURRY FEED (DSSF) GROUT VAULTS

G. A. Whyatt

March 1991

Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington 99352

9 2 1 2 4 1 1 0 9 0 6

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

9 2 1 2 4 1 4 0 9 0 7

SUMMARY

The low-level fraction of liquid, radioactive wastes stored in double-shell tanks on the Hanford Site will be disposed of as a grouted waste in large, subsurface concrete vaults. This report describes the potential for generation and release of gas from vaults containing double-shell slurry feed (DSSF) grout.

The majority of the radiolytically produced gas in DSSF grout is expected to be hydrogen. Because of the quantity of hydrogen that may be produced, the grout vault design must provide a means for venting. It is recommended that a passive approach to hydrogen venting be pursued to show that the hydrogen can diffuse through the diffusion barrier without the presence of a vent. Tests to measure the diffusivity of hydrogen in the solid barrier would be required to support such an approach.

If a passive vent is not feasible, it is recommended that a vent with a design life of 250 years and a minimum 0.1-in.-diameter clear opening be installed through the solid diffusion barrier of the grout vault to prevent pressurization. The gas will flow through this small vent with only small backpressures. The penetration of the diffusion barrier should have a total cross-sectional area of less than 1.2 in.^{2} to limit long-term water vapor diffusion and to minimize the impact on the performance assessment.

Based on currently available information, there is confidence that after 250 years the rate of gas generation will be slow enough so that if the vent closes, the hydrogen can be released by diffusion through the solid asphalt/diffusion barrier that surrounds the grout vault.

A potential exists for the formation of explosive mixtures of hydrogen in the porosity of the soil near the vent and in the porosity of the gravel in the catch basin of the vault. It is recommended that ignition sources be excluded from the catch basin of the vault and from the soil. Tests are recommended to determine whether the hydrogen can burn in the porosity of the gravel, and to further define the quantity and type of gas generated in the grout.

An analysis of radon generation was performed. Using conservative assumptions, the maximum annual effective dose equivalent to someone living downwind at the edge of the grout site would be 8.1E-3 mrem/yr.

An analysis of tritium release in the form of hydrogen was performed. Using conservative assumptions, the annual effective dose equivalent to a person living downwind at the edge of the grout site would be 1.8E-2 mrem/yr[a].

No need for vents in the closure cover of the vault has been identified. The quantity of gas generated is fairly small, and the gas should dissipate after escaping from the diffusion barrier. Pressurization is not a concern since the closure cover is not a sealed system.

(a) In DOE Order 5480.1A, derived concentration guidelines are provided based on a 100 mrem/yr dose under a specified exposure scenario. Although the specific exposure assumptions were not used in the dose calculation, the estimated dose is well below 100 mrem/yr.

CONTENTS

SUMMARY iii

INTRODUCTION 1

ANALYSIS OF GAS GENERATION 2

SELECTION OF A RADIOLYTIC GAS YIELD 2

DETERMINATION OF DOSE RATE 4

GAS GENERATION RATE 5

EVALUATION OF HYDROGEN MIGRATION 8

ESCAPE OF HYDROGEN FROM THE GROUT AND CONCRETE VAULT 8

DIFFUSION OF HYDROGEN THROUGH THE DIFFUSION BARRIER 9

SIZE OF THE HYDROGEN VENT 10

VENT DESIGN GUIDANCE 12

POTENTIAL EXPLOSION HAZARD 13

RADON GENERATION AND RELEASE 14

TRITIUM RELEASE 18

CLOSURE COVER VENTS 19

CONCLUSIONS 20

RECOMMENDED TESTS 22

REFERENCES 23

APPENDIX: ASSUMPTIONS AND BASES FOR SEVERAL VALUES IN REPORT

INTRODUCTION

The low-level fraction of liquid radioactive wastes stored in double-shell tanks on the Hanford Site will be disposed of in grout. The liquid waste will be mixed with dry cementitious solids and pumped into engineered concrete vaults where the mixture will solidify, thereby reducing the mobility of the waste. The concrete vault and leachate collection basin below the vault will be completely encased in a solid asphalt/aggregate barrier or cocoon designed to prevent ionic diffusion of contaminants to the soil and to prevent vapor diffusion of water from the soil to the waste. Additional barriers will be placed over the top of the system to reduce advection of water in the soil surrounding the vault.

The grout considered in this report is produced by mixing double-shell slurry feed (DSSF) with a dry blend of materials consisting of 28% blast furnace slag, 28% class F fly ash, 4% type I,II Portland cement, and 40% calcium carbonate. The waste has a high pH and contains a high nitrate (NO_3^-) concentration. The majority of the radioactivity in the waste results from ^{137}Cs decay. Radiolysis of grout components will produce gases, potentially causing pressurization or other problems. The primary gas produced is expected to be hydrogen. Because tritium (^3H) exists in the waste, venting of hydrogen gas provides a potential pathway for the release of radioactive ^3H from the vault to the surface. In addition, the radioactive gas radon is produced through the decay chain of uranium. Radon provides another potential pathway through which radiation could reach the surface. These potential problems are evaluated and discussed in this report.

The report begins with a description of the analysis completed in order to estimate the gas generation rate in the grout. The impacts of hydrogen generation and radon and tritium release are then discussed. Next, the need for vents in the closure cover is evaluated. Conclusions from the analysis are then presented. Finally, tests which would provide useful information for the design of the disposal system are recommended.

ANALYSIS OF GAS GENERATION

SELECTION OF A RADIOLYTIC GAS YIELD

A key factor in a gas generation analysis is the gas generation rate. Previous work on the radiolytic generation of gas was examined to select a reasonable gas generation rate for the work reported here. Normally, the yield of a gas (or other product) from radiation is expressed as a "G" value, which is the number of molecules of gas produced per 100 electron-volts of energy absorbed. In order to make the results easier to interpret in terms of the grout vault, an equivalent term was used with the units (moles of gas produced)/[(grams of material)(dose in rad received)].

A review of the literature identified several previous experiments in which the radiolytic production of gas was examined. Matherson and Ritter (1971) irradiated water using ^{60}Co to determine the gas generation rate. The rate of hydrogen generation in pure water was approximately 4.51E-13 mol/(g H_2O rad). When nitrate solutions >1.3 molar were irradiated, the hydrogen generation rate decreased to 1.385E-14 mol/(g soln rad).

Work by Gray and Simonson (1985) indicates a value of 7.14E-13 mol/(g H_2O rad) for gamma radiation of water. Gray and Simonson also measured the rate of gas generation from alpha irradiation of water and found it to be 2.32E-12 mol/(g H_2O rad). In pure water, oxygen is produced in a stoichiometric ratio along with the hydrogen. As the mixture becomes more complex, the reactions become more complex.

Hyder (1965) measured the rate of oxygen generation in water with nitrate concentrations of 1E-3 molar to 4 molar and pH between 7 and 13. Elevated pH tended to increase the conversion of nitrate to nitrite, resulting in the release of oxygen. A tracer study using ^{18}O determined that up to 39% of the oxygen released from the solution is from nitrate at 4 molar concentration. Lower concentrations result in a lower fraction of the oxygen being generated from nitrate. The total oxygen generation rate was up to 3.9E-12 mol/(g soln rad). Hydrogen generation was not discussed.

Three articles were located in which the generation of gases in mortars was reported. Lewis and Warren (1989) studied the rate of gas generation

from mortars consisting of Portland cement, class F fly ash, and ground blast furnace slag. Red clay and glass beads were added to some mortars. The study examined the effect of dry constituents, KCl content, temperature of irradiation, and additives CaS and FeS. It was also found that, in any mix containing blast furnace slag, oxygen was not produced. In addition, oxygen that was initially in the air above the grout was consumed. Oxygen was produced when slag was not present, but was eliminated when CaS or FeS was added to the mix. It was found that an elevated temperature (120°C) decreased the rate of gas generation. It was found that Cl^- can increase the hydrogen generation rate, but, at an elevated temperature, the rate is reduced by up to a factor of 7. The greatest reported hydrogen generation rate was 3.65E-13 mol/(g mortar rad).

A second article by Bibler (1978) examined numerous mortar compositions. The compositions most applicable to the grout vault consisted of Fe_2O_3, NO_3, NO_2, water, and cement. These mortars were exposed to 8.9E4 rad/h of gamma radiation at 41°C. It was found that oxygen was consumed and not produced. Hydrogen was produced at a rate of 1.45E-13 mol/(g mortar rad). A small amount of N_2O was also produced. The rate of N_2O production was dependent on the nitrite concentration in the mortar. Additional tests using alpha radiation found that over a long time period, both oxygen and hydrogen were produced in a ratio of 0.5:1. Equilibrium pressures at a dose rate of 2.8E7 rad/h for mortar containing Fe_2O_3 and MnO_2 were measured at 120 and 66 psi, respectively.

In a report by Friedman et al. (1985), the grout studied was produced from a dry blend consisting of 38.5% type I-II Portland cement, 38.5% Class "F" fly ash, 7.7% Indian red pottery clay (IRPC) and 15.3% attapulgite. This formulation is very similar to the formulation used for phosphate/sulphate waste (PSW) grout. The waste used for the grout was a simulant for double-shell slurry (DSS) consisting of 1.1 M $NaAlO_2$, 2.6 M NaOH, 2.0 M $NaNO_3$, 1.6 M $NaNO_2$, 4.8 g/L of TOC and several minor components in concentrations <0.1 M. The grout, consisting of 42 wt% dry blend and 58 wt% DSS simulant, was exposed to 8E5 rad/h of gamma radiation for periods up to 11 days. The gas generated from one test consisted of 24% hydrogen, 63% N_2O, and 13% N_2. The total gas yield was 3.0E-14 mol/(g rad). Tests using alpha radiation produced primarily

hydrogen, with smaller amounts of N_2O. Oxygen and nitrogen were either consumed or produced, depending on the length of the test.

The gas generation rates obtained from the literature for various conditions are summarized in Table 1. A conservative estimate for the rate of hydrogen generation while gamma radiation dominates the dose was selected as 3.7E-13 mol/g rad. For this analysis it was assumed that oxygen will not be produced in the grout since the dry blend contains 28% blast furnace slag. The N_2O production was ignored. The production of N_2O is influenced by the presence of nitrate and nitrite in the waste, as well as by the dry blend composition. It is possible that N_2O will be produced. However, with the exception of the rates reported by Friedman, the rate of N_2O generation was much less than the rate of hydrogen generation. If the results from the Friedman report are representative of the gas produced, then the total gas generated will be somewhat less than the selected generation rate. In the work by Bibler (1978), it was found that the rate of hydrogen generation decreased with the addition of nitrates. There is a reasonable chance that the actual generation rate may be lower than the value assumed. Since the value selected is more than 50% greater than the number from Bibler for alpha irradiation of mortar, and exceeds the value for alpha radiation as given by Friedman et al. (1985), the gamma radiation gas yield will be used for alpha, beta, and gamma radiation.

DETERMINATION OF DOSE RATE

The radionuclide inventory for the DSSF grout was entered into a spread sheet calculation to determine the total dose from all radionuclides over time. An example of the spread sheet is shown in Table 2. The inventory was chosen as the upper-bound inventories developed for use in the performance assessment of grouted double-shell tank waste (Hendrickson 1990).
Table 2 indicates the radionuclide content and dose rate after 30 years of decay. The initial dose rate is 310 rad/h. The calculated dose rate as a function of time is shown in Figure 1. The data from this plot were generated from the spread sheet by entering different decay times and calculating the dose rate.

TABLE 1. Yields of Radiolytic Gas from Various Compositions as Reported in the Literature

mol/g rad	Summary of Conditions
4.51E-13	^{60}Co source irradiating water (Matherson and Ritter 1971)
1.39E-14	^{60}Co source irradiating 1.3 M nitrate solution (Matherson and Ritter 1971)
7.14E-13	Gamma irradiation of water (Gray and Simonson 1985)
2.32E-12	Alpha irradiation of water (Gray and Simonson 1985)
1.45E-13	Cement/Fe_2O_3 highest rate reported, mix contains no nitrate (Bibler 1978)
2.90E-14	Cement/Fe_2O_3, nitrate, nitrite - highest rate reported, mix contains ≈2 wt% NO_3, NO_2 (Bibler 1978)
1.33E-13	Cement/Fe-Mn waste, no nitrate, ^{244}Cm alpha source (Bibler 1978)
3.65E-13	Slag/fly ash/cement mix - highest rate reported (6% KCl) (Lewis and Warren 1989)
2.49E-13	DSS simulant-fly ash/cement/IRPC/attapulgite: alpha radiation, primarily hydrogen with N_2O (Friedman et al. 1985)
3.00E-14	DSS simulant-fly ash/cement/IRPC/attapulgite: gamma radiation, primarily N_2O with hydrogen. O_2 partially consumed (Friedman et al. 1985)

GAS GENERATION RATE

In the previous two sections a gas generation yield has been selected (3.7E-13 mol/(g rad)) and the dose rate has been determined over time. Initially the dose rate is 310 rad/h, after which it decreases following the decay of ^{137}Cs. To calculate the gas generation from a single vault, the yield is multiplied by the dose rate and mass of grout as follows:

$$[3.7E\text{-}13\ \text{mol}/(\text{g rad})][310\ \text{rad/h}][8.8E9\ \text{g grout}] = 1.01\ \text{mol/h}$$

TABLE 2. Spread Sheet Used for Calculation of Dose Rates Over Time

ISOTOPE	T 1/2 (HRS)	DECAY CONSTANT (HR-1)	INITIAL ACTIVITY (Ci/L)	FINAL ACTIVITY (Ci/L)	WATT PER CURIE	139.172 WATT PER LITER	5.32E+07 rad Integrated Dose, rad
3H	1.07E+05	6.48E-06	1.14E-05	0.00E+00	1.10E-04	0.00E+00	4.20E+01
14C	5.02E+07	1.38E-08	7.86E-07	7.83E-07	2.93E-04	2.29E-10	1.31E+01
60Co	4.62E+04	1.50E-05	2.00E-05	3.87E-07	1.54E-02	5.96E-09	4.36E+03
79Se	5.69E+08	1.22E-09	1.80E-05	1.80E-05	2.49E-04	4.47E-09	2.55E+02
90Sr	2.51E+05	2.77E-06	7.90E-03	3.82E-03	1.16E-03	4.43E-06	3.71E+05
90Y	6.41E+01	1.08E-02	7.90E-03	3.82E-03	5.53E-03	2.11E-05	1.77E+06
94Nb	1.75E+08	3.96E-09	2.50E-05	2.50E-05	1.02E-02	2.54E-07	5.10E+00
99Tc	1.87E+09	3.71E-10	6.40E-05	6.40E-05	5.01E-04	3.21E-08	1.83E+03
106Ru	8.84E+03	7.84E-05	1.20E-02	1.34E-11	5.95E-05	7.97E-16	1.97E+03
106Rh	8.31E-03	8.34E+01	1.20E-02	1.34E-11	9.55E-03	1.28E-13	3.17E+05
129I	1.38E+11	5.04E-12	2.20E-07	2.20E-07	4.68E-04	1.03E-10	5.87E+00
134Cs	1.81E+04	3.84E-05	3.40E-03	1.42E-07	1.02E-02	1.45E-09	1.96E+05
137Cs	2.64E+05	2.62E-06	2.60E-01	1.31E-01	1.01E-03	1.32E-04	1.08E+07
137m Ba	4.20E-02	1.65E+01	2.46E-01	1.23E-01	3.92E-03	4.84E-04	3.97E+07
238U	3.91E+13	1.77E-14	1.14E-08	1.14E-08	2.49E-02	2.84E-10	1.62E+01
239Pu	2.11E+08	3.28E-09	1.20E-06	1.20E-06	5.87E-02	7.04E-08	4.01E+03
241Am	3.79E+06	1.83E-07	1.40E-06	1.33E-06	3.28E-02	4.38E-08	2.56E+03
					Totals:	6.42E-04	5.32E+07 rad

139.172 rad/hr

year 30

Cm244 neglected (t1/2=18yr, 1.7e-7 Ci initial)
Pu 239/240 treated as 239
U234 combined with U238, (note long half lives)
U235 neglected
W/Ci for 94Nb, 79Se from Urbin Jenquin to agree with Origen Code

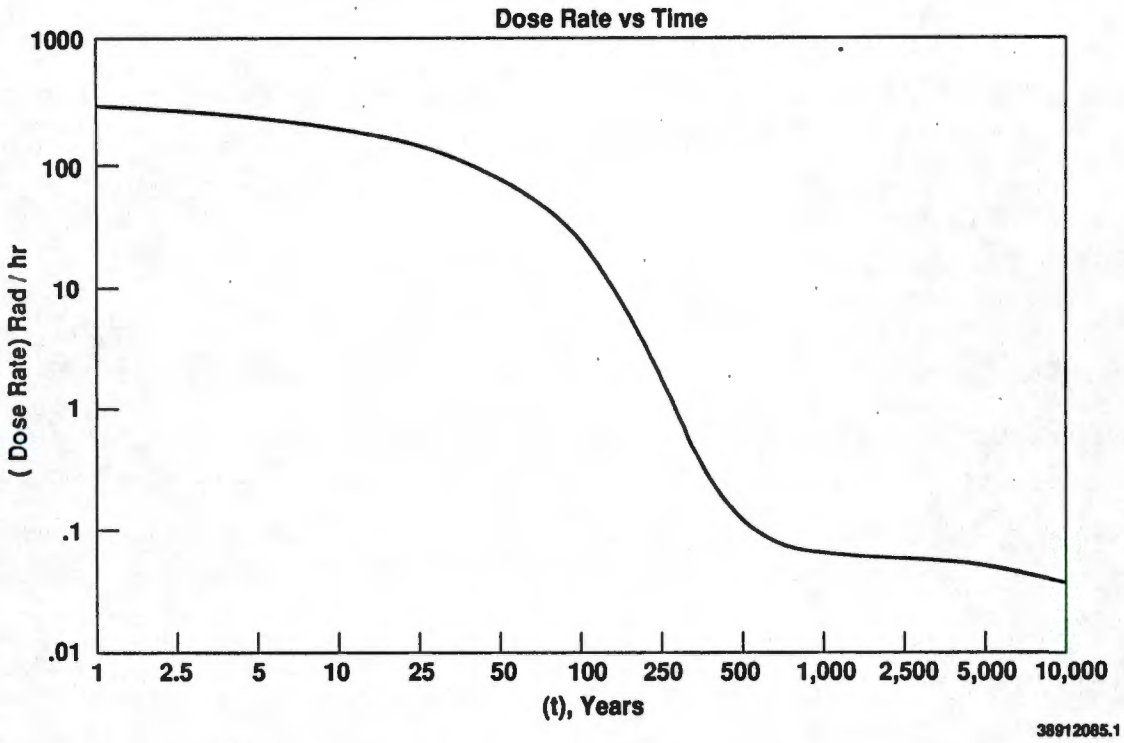


FIGURE 1. Dose Rate in Grout Over Time

9 2 1 2 4 1 4 0 9 1 7

EVALUATION OF HYDROGEN MIGRATION

ESCAPE OF HYDROGEN FROM THE GROUT AND CONCRETE VAULT

The escape of hydrogen from the grout mass itself has not been evaluated in detail. In the studies performed on concrete exposed to gamma radiation (Bibler 1978), it was found that an equilibrium hydrogen pressure is reached. The equilibrium pressure changes with mortar composition and is related to the dose rate, with higher dose rates producing higher equilibrium pressures. At dose rates of 0.1 Mrad/h, equilibrium pressures in two mortars were 20 and 40 psi, while at dose rates of 27 Mrad/h the pressures were 60 and 110 psi. In another mortar of a different composition, the gas generation was stopped at 36 psi at 0.89 Mrad/h, while at 14 Mrad/h the pressure was more than 200 psi. An alpha dose rate of 0.4 Mrad/h also produced pressures greater than 200 psi, indicating a different mechanism for gas generation from alpha radiation.

In the DSSF grout the initial dose rate will be only 3E-4 Mrad/h, which would likely result in a lower equilibrium pressure. It is possible that within the grout matrix, equilibrium pressures may be reached that will result in the production of less than the estimated quantity of hydrogen. However, the equilibrium pressure is difficult to predict due to the dependence on composition. In the event that equilibrium pressures are not reached, the entire quantity of hydrogen will diffuse through the grout and concrete and escape to the catch basin.

If the mobility of the hydrogen in the grout is too low, the pressures could potentially produce micro-cracks in the grout through which the hydrogen would easily vent. Such cracks would not have a detrimental effect on the performance of the system. Only if leaching under saturated conditions occurred would cracking have a detrimental effect. Such leaching is not expected to occur.

DIFFUSION OF HYDROGEN THROUGH THE DIFFUSION BARRIER

When the grout is poured, the gas generation rate for one vault is estimated to be approximately 1.01 mol/h. The diffusion barrier is not designed to contain gas pressures. It is likely that small imperfections introduced in the barrier during construction will allow some leakage of hydrogen under pressure. However, if it is assumed that the diffusion barrier is completely sealed and the vault becomes pressurized to 50 psi without damage, then the diffusivity of hydrogen that is required to prevent further pressurization can be calculated. This is only an order-of-magnitude estimate based on an overburden pressure on top of the catch basin of approximately 45 psi.

The transport of hydrogen through the solid barrier involves first a solubility of hydrogen in the asphalt phase and then diffusion through the asphalt. If the solubility is assumed to follow Henry's Law, then the effect of solubility can be lumped into a diffusion coefficient. Since the solubility of hydrogen in the barrier is not known, the diffusion coefficients will be calculated with the solubility effect included.

With a hydrogen pressure of 50 psi, the diffusivity must be >1E-5 cm^2/s in order to prevent further pressurization (see appendix for details). Unfortunately, data on hydrogen diffusion through asphalt are not available. For comparison, other data were obtained for other gases diffusing through asphalt. A diffusion-only value (not including solubility effects) for CO_2 was measured at 1E-5 cm^2/s (Mehrotra et al. 1987). Diffusivities of radon (including solubility effects at the interface) in different asphalts were measured at 4.4E-7 to 2.8E-6 cm^2/s (Baker et al. 1984). Therefore, in the absence of data for diffusion of hydrogen through the barrier, a small vent should be installed in the diffusion barrier to permit the escape of radiolytic hydrogen.[a]

(a) Diffusivity of hydrogen would be greater than diffusivity of CO_2 or radon. However, the observed diffusivity of the gas in asphalt will be a combination of the solubility of the gas in the asphalt (Henry's Law) and the diffusivity in the asphalt. Since the relative solubilities are not known, there is uncertainty in the comparison.

A design life of 250 years for the vent is desirable. If the vent remains open for 250 years and then closes completely, 328 years will be required to reach 50 psi, assuming no leaks and no diffusion through the barrier. At this point, the rate of gas generation will be extremely small and it is reasonable to assume that the hydrogen can diffuse through the diffusion barrier. Assuming the vent closes at 250 years and using 50 psi pressure inside the barrier, the diffusivity of hydrogen through the barrier would have to be greater than 4E-9 cm^2/s to vent the gas. It is reasonable to assume that the diffusivity of hydrogen through the barrier will not be lower than this based on comparison to the diffusivities presented in the previous paragraph.

After 500 years, the rate of hydrogen production is extremely low such that pressurization from atmospheric pressure to 50 psi will require 3300 years assuming there are no leaks, diffusion, or reactions acting as hydrogen sinks.

SIZE OF THE HYDROGEN VENT

The amount of gas generated is fairly small from the standpoint of gas flow. The estimated initial rate of gas generation for the grout is 1.01 mol/h, which is equivalent to 20.6 ft^3/d at 20°C and 1 atm pressure. The vent was sized to handle more than the maximum rate of gas generation that will occur. Two factors control the sizing of the vent: it must be large enough to allow the gas to escape without producing excessive backpressure, and it must be small enough that the rate of water diffusion through the vent over long time periods will not significantly contribute to the rate of contaminant release. The vent was nominally sized at 0.10 in. dia. for a 6-ft section passing through the diffusion barrier. The end of the vent where the gas exits to the soil is assumed to be 1-in.-dia. and filled with pea gravel.

The backpressure produced by the venting gas is affected by contributions from the frictional pressure drop in the vent line and the backpressure produced in venting to the soil. Venting of gas into soil can be described by the equation (Rolston 1986):

$$P_v = \frac{r}{4 \pi R^2} \frac{Q \mu}{k_a K}$$

Where:

P_v = back pressure [g cm/(cm^2 s^2)]
r = unit length = 1 cm
R = vent exit radius = 1.27 cm
Q = gas flux cm^3/s = 8.36 cm^3/s @ 90°C, 1 atm
μ = H_2 viscosity, 90°C = 1E-4 g/cm s (Perry and Chilton 1973)
ka = soil relative air permeability = 0.92 (Cary et al. 1989)
K = soil intrinsic permeability = 3E-9 cm^2 (Fayer et al. 1985)

This equation describes a vent discharging into an infinite mass of soil and expanding in three dimensions. When solved and converted to psi, P_v = 0.22 psi. The gas is limited in expansion in that it cannot expand in the direction of the vault. However, the backpressure is clearly not significant.

The pressure drop through the vent line can be described by the Darcy formula shown below (Crane 1986, Eq. 1-4). The Darcy equation is accurate for the flow of gases when the molar volume is assumed to be that at the inlet if the pressure drop is less than 10% of the absolute pressure at the entrance (Crane 1986, pg 1-7). In this case the flow is laminar so the friction factor is defined as 64/(Reynolds #).

$$P = \frac{(\rho\ f\ L\ v^2)}{144\ D\ 2\ g}$$

where:

P = pressure drop in psi
ρ = density in lb/ft^3
f = friction factor
L = length of pipe in feet
v = mean velocity ft/s
D = diameter of pipe, feet
g = gravitational acceleration 32.2 ft/s

The calculated pressure drop is only 2.2E-4 psi. The initial pressure drop could be slightly higher since air will be forced out of the vent as hydrogen is produced. If the properties of air are used, the pressure drop is 0.05 psi, which is still an insignificant pressure drop.

The impact of this vent on the performance of the vault is estimated by assuming that the vent pipe has decayed, leaving a hole in the diffusion barrier. The water vapor is assumed to diffuse back through the vent under isothermal conditions at 13°C. The humidity in the catch basin is assumed to be in equilibrium with 4 M $NaNO_3$ solution. The porosity of the soil at the vent exit is assumed to be at 100% relative humidity. A diffusion distance of 3 feet is used since this distance is the minimum thickness of the barrier. All water diffusing through the vent is assumed to reach the waste concentration and immediately mix with the water drawn from a downgradient well. The contribution to release from a 1.2-in.^{2} penetration through the barrier is less than 1% of the current performance goals in the performance assessment for the disposal system. This area corresponds to the cross-sectional area of two standard 1/2-in. pipes passing through the barrier.

VENT DESIGN GUIDANCE

The proposed location of the vent would be at the end of the vault opposite the leachate collection tank and just above the lip of the concrete catch basin. The tube must be designed to maintain a 0.1-in.-dia. opening for a design life of 250 years. The wall thickness of the tube should be such that it will allow compaction of the asphalt barrier on top of the tube without collapsing the tube. Filters on the ends of the vent tube are advisable to prevent aerosol particles from settling in the tube. The environment of the vent tube is not particularly corrosive since it has asphalt outside the tube and likely will have an oxygen-starved gas flowing through it. However, care should be taken that corrosion scale does not plug the opening of the vent.

The ends of the tube can be connected to 1-in. pipe, with an abrupt entry of the tube into the pipe and a screen just beyond the tube ends to prevent clogging by soil. The pipe section should be filled with pea gravel before installation to prevent clogging of the pipe and to reduce the pressure drop in venting to the soil.

POTENTIAL EXPLOSION HAZARD

The generation of hydrogen may create flammable gas mixtures in the gravel of the catch basin and in the porosity of the soil surrounding the vault. Small amounts of N_2O may be produced by irradiation of the grout. It is uncertain whether oxygen will be produced or scavenged by reactions occurring as a result of the grout being irradiated. However, it is possible that at some time a flammable mixture of hydrogen will exist in the catch basin of the vault. Also, flammable concentrations of hydrogen may exist in the porosity of the soil near the vent. Whether or not hydrogen can burn in the porosity of the soil is unknown. It may be that the high heat capacity of the gravel or soil will prevent combustion of the hydrogen.

The best way to ensure that the hydrogen does not burn is to exclude ignition sources. Potential ignition sources should be excluded from the grout vault catch basin, leachate collection tank, and surrounding soil. If ignition sources cannot be excluded, additional study of the flammability of hydrogen in the porosity of sand and gravel and study of the type and quantity of gases produced should be performed.

RADON GENERATION AND RELEASE

Double-shell slurry feed waste contains large quantities of ^{238}U, which ultimately decays through the radioactive gas ^{222}Rn (radon). Radon has a half-life of 3.825 days. The decay process is shown in Figure 2. The decay passes through ^{234}U, ^{230}Th, and ^{226}Ra before decaying to radon. Therefore, the best estimates of radon generation would be obtained from the concentration of these isotopes.

Information was not available on the concentration of ^{230}Th or ^{226}Ra in the waste. Lacking any information with which to estimate the radon generation rate, the radon generation was assumed to be in equilibrium with ^{234}U decay. This assumption is conservative since most of the ^{230}Th and ^{226}Ra is removed in processing the uranium ore.

The quantity of radon gas that will exist at any one time in equilibrium with the decay of ^{234}U is only 3.6E-9 moles. Therefore, from the standpoint of pressurization, the volume of radon is negligible. In the absence of a vent or if the vent eventually closes over a long period of time, radon release from the vault will be negligible. The diffusivity of radon through an asphalt hydraulic mix has been measured at 3E-6 cm^2/s (Baker et al. 1984). The asphalt hydraulic mix that was used was very similar to the material used to construct the diffusion barrier surrounding the grout vault. Therefore, diffusion through the diffusion barrier will be slow enough that the radon will decay before escaping the vault. However, with a vent that allows release of radiolytic hydrogen, the radon can escape from the vault system to the soil where it will then diffuse to the surface. An attempt has been made to quantify the radon release in a conservative calculation to eliminate concern over this issue.

Equations have been developed to estimate the release of radon from uranium mill tailings piles (Rogers et al. 1980). In order to estimate the amount of radon that might escape from the vault, the surfaces on the bottom and sides of the grout were assumed to represent the surface of an infinitely

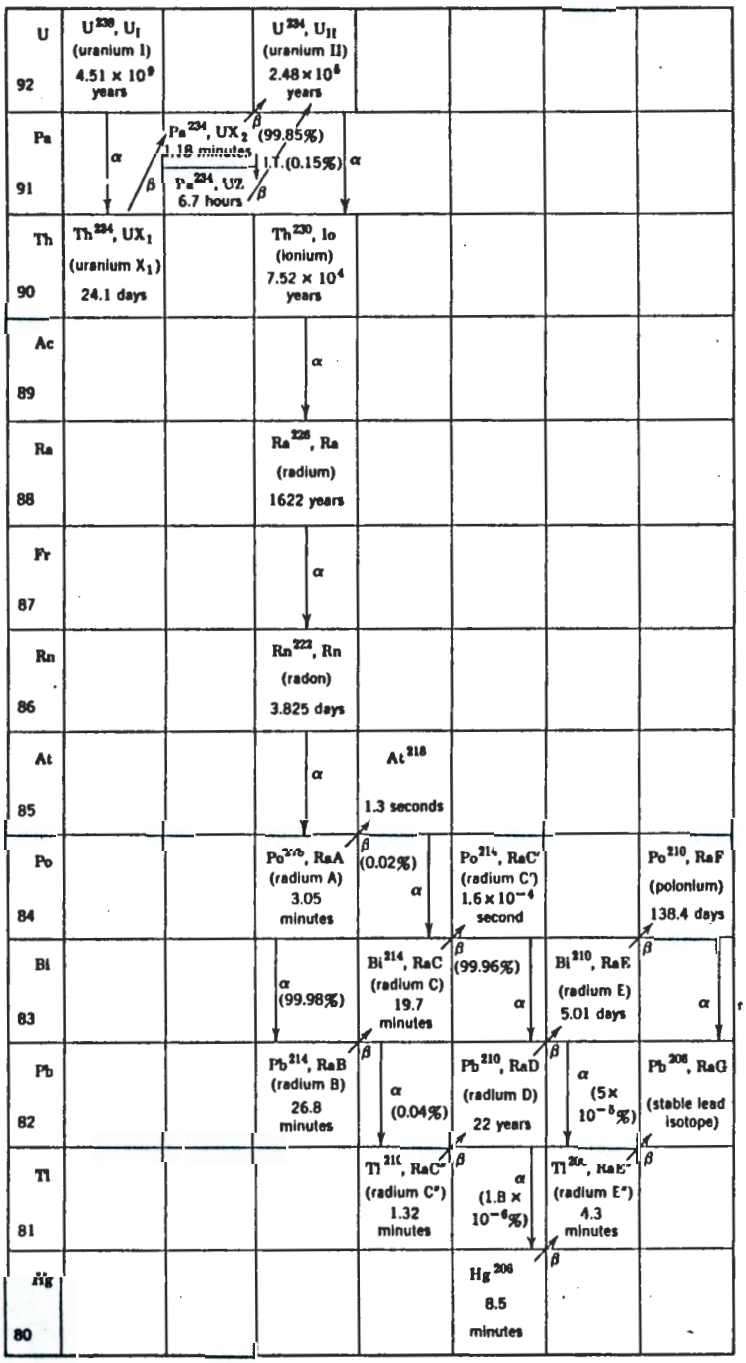


FIGURE 2. Decay Chain of Uranium (from Friedlander et al. 1964)

deep uranium mill tailings pile. For purposes of calculation, the transport within the grout was assumed to be similar to that in a soil used as backfill for the vault (porosity = 0.38). The equation that describes release from a tailings pile is shown below (Rogers et al. 1980).

$$J_0 = \frac{D_e (S - C_0 a^2)}{a}$$

Where:

J_0 = radon flux (pCi/ m^2 s)
C_0 = surface concentration (assume ≈0)
D_e = effective diffusivity = Dp, p = porosity = 0.38
radon in air = 0.12 cm^2/s
= 4.56E-6 m^2/s
a = $(L\ p\ /\ D_e)^{0.5}$
L= decay constant for radon = 2.10E-6 s^{-1}
= 0.4183 m^{-1}
S = $(R\ rho\ L\ E)/D_e$
R = radium activity per gram of grout = 13.86 pCi/g
rho = density of grout = 1.66E6 g/m^3
E = emanating power: fraction of radon that escapes from grains of matrix. Can vary from 0.01 to 0.91 for ores. Typical value is 0.20.
E = 0.20.

When J_0 is calculated and multiplied by the area of the bottom and sides of the grout, 38,946 pCi/s of radon is released from the grout. This quantity represents approximately 15% of the total radon generated within the grout. The diffusivity of radon through concrete is on the order of 2E-5 cm^2/s (Rogers et al. 1980), which would greatly reduce radon release from the vault. However, it is possible that the radon could be carried by radiolytic hydrogen through small cracks in the concrete. Therefore, no credit has been taken for the concrete vault.

The volume of the vapor space of the catch basin is 2225 ft^3 (based on KEH design analysis #WO ER9089 with gravel porosity = 0.26). The maximum hydrogen generation rate is approximately 26 ft^3/d (90°C, 1 atm). If there was not a gas vent and the radon reached an equilibrium concentration, it would reach a concentration of 8.34E6 pCi/ft^3. The 26 ft^3/d of hydrogen is assumed to exit the gas vent with this concentration of radon gas and enter

the soil, thus providing a source of radon of 2508 pCi/s at the exit of the vent.

The vent is assumed to be located on the end of the vault. The spacing between ends of vaults is assumed to be 40 feet. The gas is assumed to spread to the width of the vault only so that the flux occurs over an area of 186 m^2 (40 x 50 ft). The selection of an area will affect only the flux per unit area at the surface, and not the total release of radon to the air or the calculated dose. The flux of radon through cover material has been studied (Rogers et al. 1980); an equation describing this flux is shown below.

$$J=J_0 \exp[-(L\ p/D_a)^{0.5}\ b]$$

where:

J = flux from surface of the soil pCi/(m^2 s)
J_0 = flux into cover material, in this case from the vent
= (2508 pCi/s)/186 m^2 = 13.48 pCi/(m^2 s)
b = cover soil thickness = 17 m
D_a = D_e h(b): h(b)≈1.0 for thick soil covers
J_b : (for definition see Rogers et al. 1980)
(other notation is the same as the previous equation)

When calculated, this gives a flux at the soil surface = 1.1E-2 pCi/m^2 s. This value will decrease with the half-life of ^{137}Cs since the hydrogen gas is required to carry the radon through the vent. If all 44 vaults are assumed to be poured with a site area of 2033 m^2 per vault, and all vaults are releasing at the maximum rate, the radon emission for the site is 1.01E-3 pCi/(m^2 s). The dose was estimated using the GENII code assuming that radon is in equilibrium with its daughter products and that the wind travels along the long dimension of the site (442 m) at a speed of 1 m/s with mixing in the air occurring to a height of 1 m. For a person living downwind at the edge of the site, the annual effective dose equivalent for inhalation and external exposure is 8.1E-3 mrem/yr. Therefore, the release of radon is not a concern[a].

(a) In DOE Order 5480.1A, derived concentration guidelines are provided based on a 100 mrem/yr dose under a specified exposure scenario. Although the specific exposure assumptions were not used in the dose calculation, the estimated dose is well below 100 mrem/yr.

TRITIUM RELEASE

The DSSF waste contains tritium that will be converted to a hydrogen gas by the radiolysis of tritiated water. The upper bound inventory of tritium in each vault is 59 Ci (6.08E-3 g). Assuming that tritium and hydrogen gases are produced at the same rate by radiolysis, 1.021E-5 Ci/d of tritium as hydrogen gas will be produced in the grout. Some of this tritium will be released through the vent. Tritium containing hydrogen gas will be produced only in the early life of the vault because of the 12.3-year half-life of tritium. Over the first 30 years, the release will fall by roughly a factor of 12 due to decay of the tritium and decay of the radionuclides causing radiolysis. Tritium is not generated in the waste.

It was conservatively assumed that all of the tritium generated in the grout reaches the surface. A dose was estimated using the GENII code. GENII does not calculate dose from tritium in hydrogen gas. Therefore, the tritium was treated as tritiated water, which is conservative. Only the inhalation pathway was evaluated. It was assumed that all 44 vaults were releasing hydrogen at their maximum rate. A wind speed of 1 m/s travelling along the longest dimension of the site (442 m) with mixing occurring to a height of 1 m was assumed. The annual effective dose equivalent resulting from tritium release to a person living downwind at the edge of the site is 1.8E-2 mrem/yr. Therefore, tritium release should not be a problem.

CLOSURE COVER VENTS

No need for vents in the closure cover has been identified. The hydrogen expected to be released from the vent in the diffusion barrier is only about 20.6 ft^3/d. This quantity of hydrogen should easily disperse in the soil. Pressure cannot build under the closure cover since it is not a sealed structure. Vents would be required only if pressurization was a potential issue.

CONCLUSIONS

The conclusions of this analysis are as follows:

1. The rate of gas generation is expected to be approximately 3.7E-13 mol/(g rad). The gas is expected to be primarily hydrogen, but O_2 and N_2O may also be produced. The yield of gas (per rad) is expected to be roughly the same after alpha and beta radiation dominate the dose.

2. The maximum dose rate in the grout is 310 rad/h when the grout is first poured. The dose rate and gas generation follows the decay of ^{137}Cs until approximately 500 years, after which the dose is controlled by alpha and beta radiation. A breakdown of radionuclides was provided in Table 2.

3. It is possible that equilibrium gas pressures will be reached inside the grout matrix, which will decrease the amount of hydrogen generated. If equilibrium is not reached and the mobility of the hydrogen is insufficient to relieve pressure, microcracks in the grout might open to release the pressure. These cracks will not affect the performance of the grout disposal system.

4. It is recommended that a passive approach to venting hydrogen be pursued. It is possible that the hydrogen will diffuse through the barrier without a vent. Measurements of hydrogen diffusion through the solid asphalt barrier would be needed to support a design that does not include a vent. Also, the gas composition and generation rate for the specific waste and dry blend being used may need to be measured.

5. Based on currently available information, the rate at which hydrogen diffuses through the diffusion barrier may not be high enough to allow the hydrogen to escape during the first 250 years. There is reasonable confidence that, after 250 years, the hydrogen generation rate will decrease to a level that is low enough to permit escape of hydrogen from the vault by diffusion through the barrier.

6. If a passive approach is not feasible, a vent with a 250-year design life and a 0.1-in.-diameter clear opening is recommended for venting radiolytically generated hydrogen to the soil. It is also recommended that the cross-sectional area of the penetration of the asphalt barrier be less than 1.2 in^2.

7. Flammable mixtures of hydrogen may exist in the catch basin and in the soil near the vent exit during the life of the vault. The potential for hydrogen to burn in the porosity of the soil or gravel is unknown. In the absence of further information, ignition sources should be excluded from the vault catch basin, leachate collection tank, and surrounding soil.

8. Radon generated in the vault will decay to low levels before it reaches the surface. The release of radon is not a concern.

9. In the early life of the vault, approximately 1.02E-5 Ci/d of tritium in the form of hydrogen gas may be released to the soil by the vent. The quantity of tritium released is not a concern.

10. There is not a need for vents in the closure cover.

RECOMMENDED TESTS

The following tests could be conducted to provide useful information for the design of the disposal system. Each experiment is described along with the use for the resulting data.

1. Hydrogen diffusion tests should be performed to determine the hydrogen diffusion rate through the solid asphalt/aggregate barrier. This data could support a design using a passive approach to venting the radiolytically produced hydrogen. The test data might also change the required design life requirement for vents installed through the diffusion barrier.

2. The gas generation from DSSF grout should be studied using a representative waste and dry blend to determine the type and quantity of gases produced. Tests performed at low dose rates could be used to establish equilibrium pressures in the grout. Favorable results could support a passive design approach to venting or alter the required design life of the vent.

3. The flammability of hydrogen mixtures in the porosity of a gravel bed should be studied to determine if there is a potential for explosion in the catch basin or in the soil surrounding the vault. If it is determined that the hydrogen cannot burn in these media, the requirement for excluding ignition sources from the catch basin of the vault would be removed.

REFERENCES

Baker, E. G., J. N. Hartley, H. D. Freeman, T. E. Gates, D. A. Nelson, and R. L. Dunning. 1984. Asphalt Emulsion Radon Barrier Systems for Uranium Mill Tailings - An Overview of the Technology. PNL-4840, Pacific Northwest Laboratory, Richland, Washington.

Bibler, N. E. 1978. Radiolytic Gas Production from Concrete Containing Savannah River Plant Waste. DP-1464, E. I. du Pont de Nemours and Company, Savannah River Laboratory, Aiken, South Carolina.

Cary, J. W., C. S. Simmons, and J. F. McBride. December 1989. "Permeability of Air and Immiscible Organic Liquids in Porous Media." Water Resources Bulletin 25:6(1205-1216).

Crane Company. 1986. "Flow of Fluids Through Valve Fittings and Pipe." Technical Paper No. 410, Crane Co., New York, New York.

Fayer, M. J., W. Conbere, P. R. Heller, and G. W. Gee. 1985. Model Assessment of Protective Barrier Design. PNL-5064, Pacific Northwest Laboratory, Richland, Washington.

Friedlander, G., J. W. Kennedy, and J. M. Miller. 1964. Nuclear and Radiochemistry. 2nd ed. John Wiley and Sons, New York, New York.

Friedman, H. A., L. R. Dole, T. M. Gilliam, and G. C. Rogers. 1985. Radiolytic Gas Generation Rates from Hanford RHO-CAW Sludge and Double-Shell Slurry Immobilized in Grout. ORNL/TM-9412, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Gray, W. J., and S. A. Simonson. 1985. "Gamma and Alpha Radiolysis of Salt Brines." In Scientific Basis for Nuclear Waste Management VIII, Vol. 44, pp. 623-630. Materials Research Society, Pittsburgh, Pennsylvania.

Hendrickson, D. W. 1990. Methods and Data for Use in Determining Source Terms for the Grout Disposal System Supporting Document WHC-SD-WM-TI-335, Rev.1, Westinghouse Hanford Company, Richland Washington.

Hyder, M. L. 1965. "The Radiolysis of Aqueous Nitrate Solutions." J. Phys. Chem. 69:2.

Lewis, M. A., and D. W. Warren. 1989. Gas Generation from the Irradiation of Mortar. CONF-890488--8, DE89 013280, Argonne National Laboratory, Argonne, Illinois.

Matherson, W. E., and G. L. Ritter. 1971. Process Specifications and Standards, Uranium-Plutonium Processing the PUREX Plant. ARH-2012, Atlantic Richfield Hanford Company, Richland, Washington.

Mehrotra, A. K., A. Garg, and W. Y. Svrcek. 1987. "Prediction of Mass Diffusivity of CO_2 into Bitumen." Can. J. Chem. Eng. 65:826-832.

Perry, R. H., and C. H. Chilton. 1973. Chemical Engineers' Handbook. 5th ed. McGraw-Hill Book Company, New York, New York.

Rogers, V. C., R. F. Overmyer, K. M. Putzig, C. M. Jensen, K. K. Nielson, and B. W. Sermon. 1980. Characterization of Uranium Tailings Cover Materials for Radon Flux Reduction. NUREG/CR-1081, FBDU-218-2, Prepared for the U.S. Nuclear Regulatory Commission by Argonne National Laboratory, Argonne, Illinois.

Rolston, D. E. 1986. "Gas Flux and Air Permeability." In Methods of Soil Analysis, Part 1, Agronomy Monograph 9. 2nd ed., Chapters 47 and 48, pp. 1103-1136. Amer. Soc. of Agronomy-Soil Sci. Soc. Am., Madison, Wisconsin.

APPENDIX

ASSUMPTIONS AND BASIS FOR VALUES IN REPORT

9 2 1 2 4 1 4 0 9 3 5

Below are listed several values from the report with a description of how the values were determined.

3.7E-13 mol/(g rad)- Radiolytic gas yield. See Table 1. Value is the largest generation rate obtained in the literature using a cementitious waste form. Value from Lewis and Warren (1989).

310 rad/h- The initial dose rate in the grout assuming upper bound inventory from the DSSF grout PA is distributed into 44 vaults of 1.4E6 gallons with a grout density of 1.66, and assuming that all radiation is absorbed in the grout.

1.01 mol/h- The initial rate of hydrogen generation. Value calculated = 3.7E-13 mol/(g rad) * 310 rad/h * 8.797E9 g

1E-5 cm^2/s- The diffusivity required to vent the initial hydrogen generation rate without a vent in the diffusion barrier. This assumes the diffusion barrier sides are 152.4 cm thick with an area of 1.1E7 cm^2 and the vault top and bottom are 96.5 cm thick with an area of 1.2E7 cm^2. The pressure in the vault is assumed to be 50 psi. Fick's law is used to determine the diffusivity required. Therefore, the concentration induced in the asphalt is implicitly assumed to have the same volumetric concentration for hydrogen as the gas contained inside the diffusion barrier. This is only a gross approximation. Therefore, the diffusivity effectively includes the effect of solubility at the gas/asphalt interface.

4E-9 cm^2/s- The diffusivity required to vent hydrogen by diffusion through the barrier if the vent closes after 250 years. The time required to pressurize the catch basin is included to determine the gas generation rate. The assumptions are the same as in the value above.

MAR 08 1991

ENGINEERING DATA TRANSMITTAL

1. EDT 122885

2. To: (Receiving Organization) Distribution

3. From: (Originating Organization) Process Laboratories and Technology

4. Related EDT No: 122877

5. Proj/Prog/Dept/Div: Engineered Applications

6. Cog/Proj Engr: T. L. Welsh

7. Purchase Order No: N/A

8. Originator Remarks: Issued for approval and release.

9. Equip/Component No: N/A

10. System/Bldg/Facility: 241-AN-106

11. Receiver Remarks:

12. Major Assm Dwg No: N/A

13. Permit/Permit Application No. N/A

14. Required Response Date: 2/28/91

15. DATA TRANSMITTED

(A) Item No.	(B) Document/Drawing No.	(C) Sheet No.	(D) Rev No.	(E) Title or Description of Data Transmitted	(F) Impact Level	(G) Reason for Transmittal	(H) Originator Disposition	(I) Receiver Disposition
1	WHC-SD-CP-TP-065		0	Tank 241-AN-106 Characterization	3	1,2		
	Revision 0			Results				

16. KEY

Impact Level (F)	Reason for Transmittal (G)	Disposition (H) & (I)
1, 2, 3, or 4 see MRP 5.43 and EP-1.7	1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

17. SIGNATURE/DISTRIBUTION (See Impact Level for required signatures)

(G) Reason	(H) Disp	(J) Name	(K) Signature	(L) Date	(M) MSIN	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp
1	/	Cog./Proj. Eng DW Hendrickson		2/28/91	R4-03						
1,2	/	Cog./Proj. Eng. Mgr. JA Voogd		2/28/91	R4-03						
1	/	QA JT Knight		2/28	T6-10						
1	/	Safety OA Halvorson		2/28/91	R3-09						
1	/	QA - MJ Horhota		2-28-91	S1-53						
1	/	JP Sloughter		2-28-91	T6-07						

18. Signature of EDT Originator: TL Welsh Date: 2/28/91

19. Authorized Representative for Receiving Organization — Date

20. Cognizant/Project Engineer's Manager — Date: 2-28-91

21. DOE APPROVAL (if required) Ltr No.
- ☐ Approved
- ☐ Approved w/comments
- ☐ Disapproved w/comments

BD-7400-172 (2/89)

9212241409937

SUPPORTING DOCUMENT

1. Total Pages 167

2. Title
Tank 241-AN-106 Characterization Results

3. Number
WHC-SD-CP-TP-065

4. Rev No.
0

5. Key Words
Grout, Test Plan, Waste, Characterization, Tank 241-AN-106

6. Author
Name: T. L. Welsh
T L Welsh
Signature
Organization/Charge Code 28100

7. Abstract

Twelve samples were obtained from different locations within Tank 241-AN-106. These samples were chemically and radiologically analyzed. The results of statistical analyses indicated that the contents were stratified, but that a composite sample, representing the tank, could be formed using equal volumes of the individual samples. The composite sample analyses provide an unbiased estimate of the contents of the tank after it has been mixed.

legends removed per request of Barbara Williams legal. KMG 3/91

This document contains the analytical results and statistical analysis of samples which characterize Tank 241-AN-106 waste composition.

APPROVED FOR PUBLIC RELEASE

8. PURPOSE AND USE OF DOCUMENT - This document was prepared for use within the U.S. Department of Energy and its contractors. It is to be used only to perform, direct, or integrate work under U.S. Department of Energy contracts. This document is not approved for public release until reviewed.

PATENT STATUS - This document copy, since it is transmitted in advance of patent clearance, is made available in confidence solely for use in performance of work under contracts with the U.S. Department of Energy. This document is not to be published nor its contents otherwise disseminated or used for purposes other than specified above before patent approval for such release or use has been secured, upon request, from the U.S. Department of Energy, Patent Attorney, Richland Operations Office, Richland, WA.

DISCLAIMER - This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or any third party's use or the results of such use of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof or its contractors or subcontractors. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

9. Impact Level 3

10. Authorized Manager's Name
D. A. Dodd
Authorized Manager's Signature

Specify Distribution Limit

EXTERNAL

11. RELEASE STAMP

RELEASED
DISTRIBUTION LIMITS
☐ INTERNAL ONLY
☐ SPONSOR LIMITED
(2) INTERNAL
DATE: MAR 08 1991
Sta #4

DOES NOT CONTAIN CLASSIFIED OR UNCLASSIFIED CONTROLLED NUCLEAR INFORMATION

Reviewing Official / ADC:
Date: 2/28/91

A-6400-073 (7/90) (EF) WEF124
Supporting Document (Cover Sheet)

9212410938

WHC-SD-CP-TP-065
Rev. 0

TANK 241-AN-106 CHARACTERIZATION RESULTS

Issued by

T. L. Welsh
Statistics Team
Process Laboratories and Technology

Prepared for

Westinghouse Hanford Company Grout Disposal Program

February 28, 1991

for the

U. S. DEPARTMENT OF ENERGY
RICHLAND OPERATIONS OFFICE
RICHLAND, WASHINGTON

This page is intentionally left blank

CONTENTS

1.0 SUMMARY 5

2.0 INTRODUCTION 5

3.0 OBJECTIVES 5

4.0 SAMPLES 6

5.0 ANALYTICAL MEASUREMENTS 6

6.0 HOMOGENEITY OF TANK CONTENTS 8

7.0 COMPOSITE SAMPLE 12

7.1 COMPOSITE DESIGN 12

7.2 COMPOSITE ANALYSIS 13

8.0 COMPARISON 15

9.0 ORGANIC ANALYSIS 19

9.1 ORGANIC SAMPLE PREPARATION 19

9.2 ORGANIC ANALYSIS RESULTS 19

10.0 REFERENCES 20

TABLES

1. Sample Locations 7
2. Tank 241-AN-106 Sample Numbers 9
3. Tank 241-AN-106 Composite Sample Numbers 14
4. Comparison of Individual Sample Mean to Composite Mean 16

APPENDICES

A. Tank 241-AN-106 Historical Results 21
B. Tank 241-AN-106 Sampling Locations 39
C. Tank 241-AN-106 Individual Sample Results 55
D. Tank 241-AN-106 Mean Sample Result by Location 83
E. Tank 241-AN-106 FPLSD Results 101
F. Tank 241-AN-106 Calculations to Determine the Composite 111
G. Tank 241-AN-106 Composite Results 127
H. Tank 241-AN-106 Organic Results 131

TANK 241-AN-106 CHARACTERIZATION RESULTS

1.0 SUMMARY

Twelve samples were obtained from different locations within Tank 241-AN-106. These samples were chemically and radiologically analyzed to determine the constituents of the waste. The results of the statistical analyses indicated that the contents were stratified, but that a composite sample, representing the tank, could be formed using equal volumes of the individual samples. The comparison of the individual sample mean to the composite sample mean indicated that the two means were not significantly different. Thus, the analyses of the composite sample provide an unbiased estimate of the contents of the tank after it has been mixed.

This document contains the analytical results and the results of the statistical analyses of the sample data. In addition, this document provides the analytical results of the composite sample which characterize Tank 241-AN-106 waste composition.

2.0 INTRODUCTION

Twelve samples from different locations in Tank 241-AN-106 were received by Analytical Systems Laboratories (ASL) personnel and prepared for analysis. The requested chemical and radiological analyses provided information about the character of the waste in Tank 241-AN-106. The analytical results were used to evaluate homogeneity of the waste inside the tank. A composite sample, representing the twelve waste samples, was used to test the analytical procedures required to determine if this waste is acceptable for processing and disposal as grouted waste.

3.0 OBJECTIVES

The objective of this document is to report the analytical data generated from the 12 waste samples obtained from Tank 241-AN-106. The analytical data was acquired in accordance with the test plan (Hammitt, 1989). The analytical data will be used to characterize the waste in Tank 241-AN-106.

An additional objective, as presented in the test plan, was to test the laboratory procedures and equipment in the hot cell. The cured grout samples, made while testing the hot cell procedures, will indicate whether or not the waste in Tank 241-AN-106 meet the requirements for processing and disposal in grout. Test results pertaining to this objective will be presented in a separate document.

4.0 SAMPLES

Existing information (see Appendix A) suggests that the contents of Tank 241-AN-106 are stratified but that interfaces between strata are not well defined. This information was used to determine the locations for the tank samples. Twelve samples were obtained from Tank 241-AN-106. The locations of the 12 samples were determined through the use of a stratified random sampling plan. The details associated with the sampling plan are given in Appendix B. The 12 locations are identified in Table 1. The information regarding riser location is given in Bordelon, 1986. The use of a stratified random sampling plan is supported by the EPA manual SW-846, "Test Methods for Evaluating Solid Waste," (EPA, 1986).

Twelve 100-milliliter samples in glass bottles from Tank 241-AN-106 were received by ASL at the 222-S Analytical Laboratory. Laboratory receipt records indicate that the samples were taken and received at the 222-S Analytical Laboratory on April 21, 1989. Each of the samples were logged in and received in the laboratory under a designated sample number. Two subsamples of each waste sample, formed in accordance with the test plan, were submitted to Analytical Chemical Services Laboratories (ACSL) for analysis. Each subsample, identified by an ASL sample number, was assigned a new sample number by ACSL. Direct waste samples were also submitted to ACSL for specific analyses. All the sample numbers are summarized in Table 2.

5.0 ANALYTICAL MEASUREMENTS

A list of the requested chemical and radiological analyses and the procedures used is provided in the test plan. An analysis for silicon was requested but not completed. Two additional analytes, carbonate and mercury, were added to the list of requested analyses. All analytical results are entered into the Laboratory Customer Communications System (LCCS) database. Ninety days after the samples have been slurped the data are transferred from the system and permanently archived. Personnel from the Technical Support Unit can retrieve the data upon request.

Table 1

Sample Locations

Tank 241-AN-106 Sample Locations				
Sample Number	Riser Number	*Radius	Angle	**Depth (inches)
1	22A	10	180°	331
2	22A	10	180°	220
3	22A	10	180°	100
4	22A	10	180°	20
5	1B	20	280°	292
6	1B	20	280°	224
7	1B	20	280°	137
8	1B	20	280°	49
9	16C	28	65°	366
10	16C	28	65°	210
11	16C	28	65°	114
12	16C	28	65°	16

*Radius is the distance in feet from the center of the tank.
**Depth is the number of inches from the bottom of the tank.

The analytical results stored in the LCCS database for the Tank 241-AN-106 samples must be multiplied by the appropriate dilution factor before the measurement is indicative of the original sample.

Some subsamples (as indicated in Table 2) were prepared by ASL according to the water dilution (Hammitt, 1989), which resulted in a dilution factor of 11. After dilution, these subsamples were submitted to ACSL for analysis. Several analyses (as indicated in Table 2) needed a direct sample.

The appropriate dilution factor was applied to each analytical result for all constituents of the 24 subsamples; the resulting values are listed in Appendix C. Agreement between duplicate samples is evaluated by computing the absolute percent difference (see Appendix C). Appendix D contains the mean value for each constituent by location. Illustrations of the mean value by location are also given in Appendix D.

6.0 HOMOGENEITY OF TANK CONTENTS

Several statistical tests were performed to determine whether or not the contents of the tank were homogeneous. A two-way analysis of variance (ANOVA) was performed for each constituent to detect differences within a section (horizontal layer of the tank) and differences between sections. The results of the ANOVA indicated significant differences at the 0.05 level of significance in the mean concentration values within sections and between sections for all analytes reported with actual values. An actual value is defined to be a value not identified with a "<" in Appendix C.

The significant differences between sections indicated that the tank contents are stratified. Significant differences within a section combined with significant differences between sections indicated that the interfaces between sections are not well defined. Tank 241-AN-106 waste contents are not homogeneous.

Table 2

Tank 241-AN-106 Sample Numbers

Receiving Laboratory Sample Number	ASL Sample Number	ACSL Sample Number
R4601	APH-4	R5232
	APH-28	R5235
	APH-75*	R6070
R4602	APH-6	R5233
	APH-32	R5238
	APH-76*	R6071
R4603	APH-8	R5234
	APH-34	R5239
	APH-77*	R6072
R4604	APH-10	R5274
	APH-36	R5278
	APH-73*	R6024
	APH-90*	R6291
R4605	APH-12	R5275
	APH-38	R5279
	APH-74*	R6025
	APH-89*	R6292

*Analyses which required a direct sample.

Note: The subsamples without a symbol were prepared by ASL according to the water dilution (Hammitt, 1989) which resulted in a dilution factor of 11. After dilution, these subsamples were submitted to ACSL for analysis. Several analyses needed a direct sample (see *).

Table 2, Continued

Tank 241-AN-106 Sample Numbers

Receiving Laboratory Sample Number	ASL Sample Number	ACSL Sample Number
R4606	APH-14	R5276
	APH-40	R5281
	APH-78*	R6073
R4607	APH-16	R5277
	APH-42	R5282
	APH-79*	R6074
R4608	APH-18	R5290
	APH-44	R5296
	APH-80*	R6076
R4609	APH-20	R5291
	APH-46	R5297
	APH-81*	R6077
R4610	APH-22	R5292
	APH-48	R5298
	APH-82*	R6078

*Analyses which required a direct sample.

Note: The subsamples without a symbol were prepared by ASL according to the water dilution (Hammitt, 1989) which resulted in a dilution factor of 11. After dilution, these subsamples were submitted to ACSL for analysis. Several analyses needed a direct sample (see *).

9 2 1 2 4 1 4 0 9 4 8

Table 2, Continued

Tank 241-AN-106 Sample Numbers

Receiving Laboratory Sample Number	ASL Sample Number	ACSL Sample Number
R4611	APH-24	R5293
	APH-85	R6067
	APH-50	R5299
	APH-86	R6068
	APH-83*	R6079
R4612	APH-26	R5294
	APH-52	R5300
	APH-84*	R6080

*Analyses which required a direct sample.

Note: The subsamples without a symbol were prepared by ASL according to the water dilution (Hammitt, 1989) which resulted in a dilution factor of 11. After dilution, these subsamples were submitted to ACSL for analysis. Several analyses needed a direct sample (see *).

A one-way ANOVA, using each sample location as a class, was performed to provide more detail about Tank 241-AN-106 contents. The results from the ANOVA for each constituent indicated significant differences between the 12 sample means at the 0.05 level of significance. Since significant differences existed, a multiple comparison procedure known as Fisher's Protected Least Significant Difference (FPLSD) was used to determine which sample means were significantly different. The FPLSD results are given in Appendix E.

In general, the FPLSD results showed that the waste consisted of two layers with an interface between depths 100 and 200 inches from the bottom of the tank. This region is not well defined due to an intermingling of the liquid from the two layers. Process history of Tank 241-AN-106 contents also indicates that any interface region would be in this area.

7.0 COMPOSITE SAMPLE

According to the test plan, a single composite sample was to be made from the 12 original samples.

7.1 COMPOSITE DESIGN

The results of the statistical analyses were used to develop the composite sample formulation. The calculations from which the composite formulation was developed are provided in Appendix F. The overall mean tank concentration and its associated variance or standard deviation was computed for each analyte reported with actual values using the following methodologies:

a. The stratified sampling plan was used which partitioned the tank into four sections.

b. The tank was divided into two layers, but the original stratification was taken into account. This was done for various divisions between 100 and 200 inches from the bottom of the tank.

c. The tank was assumed to be divided into two layers. The sample locations were treated as random selections within each layer. The calculations were performed using various divisions between 100 and 200 inches from the bottom of the tank.

d. The sample locations were treated as random selections from the entire tank.

The same sample locations obtained using the stratified sampling plan (four sections) could have been obtained using total randomization within the two layers or within the tank.

Statistical tests comparing the overall mean tank concentration values from each of the methodologies were evaluated using the smallest variance from all the calculations. This is considered to be conservative. The results of the statistical tests indicated that no significant differences existed between the estimates of the overall mean tank concentration. Consequently, Tank 241-AN-106 contents can be represented by compositing an equal volume of each of the 12 samples.

7.2 COMPOSITE ANALYSIS

The composite sample was made on November 21, 1989, according to the steps given in Claghorn, 1989. Six subsamples from the composite sample using different dilutions were submitted to ACSL for analysis.

Some subsamples (as indicated in Table 3) were prepared by ASL according to the water dilution (Hammitt, 1989), which resulted in a dilution factor of 11. Some analyses, upon request of the chemist, did not require as much dilution. Therefore, some subsamples were prepared with a water dilution resulting in a dilution factor of 2 (as indicated in Table 3). Several analyses (as indicated in Table 3) needed a direct sample.

The ASL sample numbers and the assigned ACSL sample numbers are given in Table 3. The list of requested analyses and the procedures used for the analyses is provided in the test plan. The analysis for silicon was requested but not completed.

All analytical results are stored in the LCCS database. The data can be obtained upon request. Each analytical result from the LCCS database for these samples must be multiplied by its dilution factor before the measurement is indicative of the original composite sample. The composite sample data are listed in Appendix G.

Table 3

Tank 241-AN-106 Composite Sample Numbers

ASL Sample Number	ACSL Sample Number	Dilution Factor
APH-64	R5850	11
APH-65	R5851	11
APH-68	R5854	1
APH-69	R5855	1
APH-66	R5858	2
APH-67	R5859	2

Note: Some subsamples were prepared by ASL according to the water dilution (Hammitt, 1989) which resulted in a dilution factor of 11. Some analyses, upon request of the chemist, did not require as much dilution. Therefore, some subsamples were prepared with a water dilution resulting in a dilution factor of 2. Several analyses needed a direct sample. These are indicated above with a dilution factor of 1. After the dilution by ASL, the subsamples were submitted to ACSL for analysis.

8.0 COMPARISON

The average results for the individual sample results and the composite sample analyses are summarized in Table 4. For these data, the means calculated from the 12 individual sample results are arithmetically the same regardless of whether the means are computed using the stratified sampling scheme or treated as observations from a random sample of the entire tank.

The standard deviations of the mean included in Table 4 were calculated: 1) using the original stratified sampling scheme and 2) assuming the observations were obtained using a randomized sampling scheme for the entire tank. The standard deviation of the mean is equal to the standard deviation divided by the square root of n, the number of observations used in determining the standard deviation.

For the original stratified sampling plan, n is equal to 3 (the number of observations within a section). For the randomization sampling plan, n is equal to 12 (the number of observations assuming the tank is one layer). As is evident from Table 4, stratification reduced the variance and standard deviation. This reduction in variance is the reason stratified random sampling plans are used.

An ANOVA was performed to compare the individual sample mean and the composite sample mean. None of the ANOVA results indicated that the individual sample mean was significantly different from the composite mean at the 0.05 level of significance. The F-test of the ANOVA was calculated using the variance from the stratified sampling scheme. The degrees of freedom associated with the sample variance from the stratified sampling plan were calculated using Satterthwaite's approximation.

The individual sample mean and the composite sample mean were also compared using the Kruskal-Wallis nonparametric test. The Kruskal-Wallis test does not assume that the data are from a normal population. The Kruskal-Wallis test is used to determine if the populations are identical. None of the Kruskal-Wallis results indicated that the two populations were significantly different at the 0.05 level of significance.

Table 4

Comparison of Individual Sample Mean to Composite Mean

Tank 241-AN-106 Inorganic Chemical and Radionuclide Summary Results					
Constituent	Units	Individual Sample Mean	Stratified Standard Deviation (Mean)	Sample* Standard Deviation (Mean)	Composite Mean
Aluminum	mg/L	9590	1710	2550	9280
Arsenic	mg/L				0.072
Barium	mg/L	< 11.1			< 11.1
Bismuth	mg/L	< 145			< 145
Cadmium	mg/L	< 49.6			< 49.6
Calcium	mg/L	77.3	14.3	19.7	90.2
Chromium	mg/L	569	95.4	144	564
Copper	mg/L	< 3.79			< 3.75
Iron	mg/L	< 8.47			< 6.90
Lead	mg/L	< 460			< 460
Magnesium	mg/L	< 2.19			2.78
Manganese	mg/L	< 55.6			< 55.7
Mercury	mg/L	< 0.05			< 0.05
Molybdenum	mg/L	< 67.8			< 66.6
Phosphorus	mg/L	6270	772	1050	6110
Potassium	mg/L	1020	144	230	1080

* Assumes total randomization within the tank.

9 2 1 2 4 1 4 0 9 5 4

Table 4, Continued

Comparison of Individual Sample Mean to Composite Mean

Tank 241-AN-106 Inorganic Chemical and Radionuclide Summary Results					
Constituent	Units	Individual Sample Mean	Stratified Standard Deviation (Mean)	Sample* Standard Deviation (Mean)	Composite Mean
Selenium	mg/L				0.134
Silver	mg/L	< 3.70			< 3.63
Sodium	mg/L	89300	10100	16300	90300
Titanium	mg/L	< 3.57			< 3.51
Uranium	g/L				0.004
Zinc	mg/L	< 9.64			< 9.44
Zirconium	mg/L	< 28.2			< 27.8
Ammonia	mg/L				123
Carbonate	M	0.350		0.065	0.327
Chloride	mg/L	2510	401	541	2460
Cyanide	g/L				0.006
Hydroxide	M	0.484		0.128	0.474
Fluoride	mg/L	< 44.0			< 61.6
Nitrite	M	0.601	.010	0.149	0.644
Nitrate	mg/L	74800	12600	18700	68500
Phosphate	mg/L	17900	2240	3420	18400
Sulfate	mg/L	2570	383	449	2150
Tritium	µCi/L				3.44
Carbon-14	µCi/L				0.335

* Assumes total randomization within the tank.

9 2 1 2 4 1 4 0 9 5 5

Table 4, Continued

Comparison of Individual Sample Mean to Composite Mean

Tank 241-AN-106 Inorganic Chemical and Radionuclide Summary Results					
Constituent	Units	Individual Sample Mean	Stratified Standard Deviation (Mean)	Sample* Standard Deviation (Mean)	Composite Mean
Cobalt-60	µCi/L	< 24.5			< 9.15
Selenium-79	µCi/L				0.240
Strontium-90	µCi/L				2230
Niobium-94	µCi/L	< 38.9			< 23.0
Tc-99	µCi/L				69.2
RuRh-106	µCi/L	< 2610			< 1390
Iodine-129	µCi/L				< 0.075
Cesium-134	µCi/L	< 41.2			< 23.7
Cesium-137	µCi/L	211000	35000	53400	205000
Np-237	g/L				< 0.003
Pu-238	µCi/L				0.020
Pu-239/240	µCi/L				0.0378
Am-241	µCi/L				0.606
Curium-243	µCi/L				0.047
TOC	mg/L				3260
Water	wt%				77.6
Density	g/cm^3	1.27	0.04	0.05	1.23

* Assumes total randomization within the tank.

9 2 1 2 4 1 4 0 9 5 6

9.0 ORGANIC ANALYSIS

The composite mean for total organic carbon (TOC) was 3.26 g/L. Since the TOC concentration was over 2 g/L which was the limit specified in the test plan, a subsample of the composite was submitted for organic analysis to the 300 Area Analytical Laboratory, operated by Pacific Northwest Laboratories (PNL).

9.1 ORGANIC SAMPLE PREPARATION

The first composite sample was prepared as described in Section 7.2 and submitted for analysis to PNL on December 21, 1989. This sample consisted of equal fractions of the twelve waste samples. Due to laboratory downtime, this composite sample was discarded. A second composite sample was prepared on August 27, 1990, and shipped to PNL for analysis on August 29, 1990.

9.2 ORGANIC SAMPLE ANALYSIS RESULTS

Volatile organic analysis using EPA (EPA, 1986) protocols on highly radioactive materials created some additional problems. Samples obtained from Tank 241-AN-106 could not be used to fill other sample vials that were prepared and preserved according to the required analysis. The radioactivity of the waste did not allow such handling. Preservatives were not used in the sample bottle because the waste was not expected to contain any biological activity that might influence the results of an unpreserved sample.

In addition to not having the sample vial preserved for volatile organic analysis, the sample bottle could not be confirmed for zero headspace. Refrigeration of the sample was not possible because of the shielding requirements for the shipping container. Sample holding times exceeded those specified by EPA (EPA, 1986), because of hot cell handling requirements and laboratory availability.

These deviations are not expected to have had a serious impact on the sample results because of the history of the waste. The waste in Tank 241-AN-106 was processed through the evaporator and stored for a number of years at temperatures at or above ambient conditions with forced ventilation. The sample handling required because of the radioactivity of the waste did not cause the sample to be subjected to a more rigorous environment for volatile component removal than was present in the tank.

The results from the speciated organic analyses are listed in Appendix H.

REFERENCES

Bordelon, M. V., 1986, SD-RE-TI-093, Rev. 1, "Double-Shell Underground Waste Storage Tanks - Riser Survey," December 8, 1986.

Claghorn, R. D., 1989, "Test Plan for the Mixing of Waste from Tank 241-AN-106," SD-WM-TP-069, Rev. 0, Westinghouse Hanford Company, Richland, Washington, August 31, 1989.

EPA, 1986, "Test Methods for Evaluating Solid Waste," SW-846, Third Edition, U.S. Environmental Protection Agency.

Hammitt, A. P., 1989, "241 AN-106 Characterization and Grout Formulation Hot Cell Test Plan," SD-WM-TP-061, Rev. 1, Westinghouse Hanford Company, Richland, Washington, June 22, 1989.

Malone, L. C. and Ostle, B., 1988, "Statistics in Research," Fourth Edition, Iowa State University Press, Ames, Iowa.

9 2 1 2 4 1 4 0 9 5 9

APPENDIX A

HISTORICAL RESULTS

TANK 241-AN-106

This page is intentionally left blank

From: Analytical Systems Laboratories
Phone: 3-1951 MO-037/200 West Area
Date: July 21, 1987
Subject: 106-AN ANALYTICAL RESULTS AND SIMULATE DEVELOPMENT

65452-87-198

To: WG Richmond 2750E/200E 3-3416

cc: DA Dodd MO-037/200W 3-2154
TV Rebagay MO-037/200W 3-1571
JK Watts MO-037/200W 3-4203
LB MO-037/200W

References:

(1) Internal Letter #65641-87-004, WG Richmond to WI Winters, "Analysis of 106-AN Samples," January 15, 1987.

(2) Internal Letter #65452-87-022, WI Winters to WG Richmond, "Analysis of 106-AN Samples," January 28, 1987.

(3) Internal Letter #65641-87-047, WG Richmond to DA Dodd, "Formulation Testing/Verification Plan for 106-AN Waste," March 27, 1987.

(4) Internal Letter #65641-87-061, WG Richmond to JE Van Beek, "Updated Schedule for Tank 106-AN Formulation Verification," April 15, 1987.

(5) Internal Letter #65641-87-063, WG Richmond to TV Rebagay, "Dry Blend Formulations to be Tested Using the Tank 106-AN Simulant," April 17, 1987.

(6) Internal Letter #65452-87-082, WI Winters to WG Richmond, "106-AN Waste Simulation," April 6, 1987.

(7) Internal Letter #65641-87-060, WG Richmond to WI Winters, "Simulant Composition for 106-AN Waste," April 14, 1987.

(8) Internal Letter #65452-87-110, WI Winters to WG Richmond, "106-AN Simulated Waste Formulation," May 6, 1987.

(9) Internal Letter #65452-87-109, WI Winters to S. Kelly, "Preparation of Synthetic 106-AN Waste for Grout Testing," May 5, 1987.

Hanford Operations and Engineering Contractor for the US Department of Energy

This letter contains two parts: Part A summarizes the analytical results from analysis of four 106-AN samples taken at different depths and their composite; Part B summarizes the procedures used to make a 106-AN simulated waste and the final analysis of that simulate.

W. I. Winters

W. I. Winters, Chemist
Analytical Systems Laboratories

WIW/cse

Attachments

PART A

106-AN SAMPLE ANALYSIS

Four samples of 106-AN at different depths were received and analyzed in accordance with References 1 and 2. The sample were sent to the 222S laboratory on February 19, 1987, in heated pigs and stored under heat lamps. When it was learned the tank temperature was approximately 70°F, the same as the laboratory temperature, the samples were removed from the heat lamps and placed in mini pigs. The visual and radiation level measurements were done at that time.

All four samples were extremely clear for waste samples with only a few particles of dark solids scattered on the bottom that looked like rust. The volume of these solids was too small to measure. Photographs taken of each sample were sent to you earlier.

A complete characterization was required only for the composited sample. The composite was prepared by stirring each sample and transferring equal 25 mL aliquots to a clean polyethylene bottle. When the composited sample was removed from the pig a large volume (approximately 50 percent) of solids was present. A large teflon stir bar was used to thoroughly stir the sample; however, the solids would not redissolve at room temperature. After the composited sample was allowed to set over the weekend the solids volume level was about 16 to 17 percent. A small sample of the needle like crystals were analyzed by polarized light microscopy and identified by Dr. D.L. Herting as Na_3PO_4 crystals in two hydrated forms. The composite was heated until the Na_3PO_4 needles dissolved at approximately 33°C. After the solids were totally dissolved, a 5 mL sample of the heated and stirred composite sample was diluted with 50 mL of deionized water. This diluted composite was submitted to Analytical Laboratories (AL) for analysis.

The solids formation behavior of the composite sample is believed to be caused by the limited solubility of Na_3PO_4 in high ionic strength (high salt concentration) solutions. In Table 1 you can see that in the upper region of the tank the phosphate level is high but the salt (Na, Al) concentration is low. As you go deeper in the tank, the phosphate concentration decreases as the salt concentration increases therefore maintaining a stable homogeneous solution. When the high PO_4^{-3} concentration upper phase was mixed with high salt concentration lower phase for the composite, the Na_3PO_4 solubility was exceeded and solids formed. This phosphate stratification could cause some engineering problems when the tank is mixed and transferred for the grout process.

This tank stratification observation led to additional analysis for the major cation and anion components on individual 106-AN samples. It also impacted the development of the 106-AN simulated waste.

The results of all the analyses are summarized in Table 1. The viscosity results were run at only one temperature, 25°C. The viscosity results are subject to a large error since the viscosity of the 106-AN is near the lower

operating limit of the equipment. The results for R2090 are estimated to be <6 cp because the standard in this region was reading high by about 60 percent. These analyses were run by B.M. Mauss of the Process Chemistry Laboratories Unit. The weight percent water was run only on the composite sample by drying at 110°C overnight and measuring the weight loss.

Radiochemical and some trace analyses were done only on the diluted composite sample. Technetium-99 (Tc-99) was analyzed by both the 222S laboratory and Pacific Northwest Laboratory (PNL) (formerly HEDL Analytical Laboratory) because earlier Rockwell Tc-99 analyses on double shell slurry were 100 times higher than those analyzed by a different PNL laboratory. Carbon-14 (C-14) analyses were done at HEDL because of problems with the present AL method and equipment. A separate wet oxidation system for C-14 needs to be developed in place of the current furnace oxidation system. Selenium-79 (Se-79) was also analyzed by HEDL since no routine method exists for the 222S laboratory. The actinides were determined by alpha counting after an anion exchange separation by Analytical Systems Laboratories Unit (ASLU).

In general, all the results increase as you go deeper into the tank except for phosphate. For some reason, possibly analytical error, the cation results for R2090 (53' 3") are lower in concentration than R2089 (44' 9"). However, the anion results from R2090 continues to increase as might be expected. This makes the cation (ICP) results on R2090 questionable. The samples or inductively coupled plasma (ICP) results for R2089 and R2090 may have been interchanged. The ICP phosphate results agree reasonably well with the ion chromatography result.

The hydroxide, OH^-, result for R2087 is very low and could be subject to a relatively large error. The gamma scans showed only cesium-137 (CS-137). Other trace gamma isotopes that may be present could not be identified because of the large dilution required to analyze for Cs-137. This large dilution would result in large less-than values for the other isotopes. Copies of the complete gamma scans are available upon request. Only three isotopes (Tc-99, Se-79, and C-14) were analyzed for all of the 106-AN samples. These analyses were performed by HEDL and copies of their reports are attached.

All of the analyses done on the composite were done on the diluted composite sample except for the actinides and the cyanide (CN^-). The values in (___) next to the composite results are the average result for the individual 106-AN analyses. Since the composite was made up of equal volumes the average and composite values should be essentially the same. For most of the major analyses (density, Al, NO_3^-, NO_2^-, and Cs-137) this was true. However, for some components, (Na and PO_4^{-3}) the agreement was poor. The average Na results were only 80 percent of the composite results. The composite result could be bias high if the final Na dilution was contaminated. However, the high Na composite value (5.43 M) results in a anion-cation balance of 96 to 97 percent which indicates it may be valid. It would require over four moles of Na to raise the average Na value to that of the composite. Reruns of the ICP analyses improved the sodium balance some, but not as well as desired. Since other analyses agree reasonably well it is assumed that the composite was properly made. Dilution of the thermally hot composite sample could result in some volumetric errors but none of this magnitude.

The hydroxide result on the composite is significantly different than the average value. The accuracy of the composite OH^- analysis could be questioned since it was run on the diluted sample which lowered its concentration to the point that aluminum (Al) may be causing a significant interference. The composite fluoride value is based on a specific ion electrode measurement which has better detection limits than the ion chromatograph (IC) method used on individual samples.

The HEDL average Tc-99 value (84.7 uCi/L) agrees reasonably well with the Rockwell composite value (96.3 uCi/L) indicating that the Rockwell method was valid for this matrix. The total beta is approximately two times higher than the sum of the other beta analysis and the Cs-137 result. The total beta is based on the beta efficiency for Co-60 which is approximately 1.5 times less than the Cs-137 beta efficiency which appears to be the major isotope in the 106-AN sample. If the total beta is calculated based on the Cs-137 efficiency about 1×10^5 uCi/L of beta activity (approximately 30 percent) is unaccounted for. The iodine-129 (I-129) analyses were performed on the diluted sample. A lower less-than value could be obtained by analyzing the direct sample without dilution. This would require pipeting a heated sample.

All of the actinide results were very low giving a high uncertainty level because of poor counting statistics. However, the transuranic (TRU) level is definitely below the 100 nCi/g limit by at least a factor of ten if you assume the Pu-236 spike is fully equilibrated with the plutonium in the sample. The plutonium analyses yields were corrected using a plutonium-236 (Pu-236) spike. Normally, the Pu-236 recovery is greater than 80 percent. However, for the 106-AN, the Pu-236 (results) were very low, only 10 to 20 percent. The sample was analyzed in duplicate twice and gave reasonable reproducible results ($\pm$ 36 percent) for these low levels of plutonium. The low recoveries are probably caused by phosphate complexing or from complexants that may be present in the 106-AN waste. Analysis of other eluent fractions of the ion exchange separation did not account totally for the Pu-236 losses. Several other experiments are planned to attempt to improve the yields. However, further fundamental studies are needed on the effects and tolerance levels of various complexants on the ion exchange separation of actinides. The americium-241 (Am-241) was measured using a CMPO/TTA extraction from the 8 M HNO_3 ion exchange eluent and the separation yields were corrected using an Am-243 spike. The Am-243 spike yields were 50 to 60 percent which is normal for this procedure. The Am-241 was analyzed in duplicate and the average value reported. The neptunium-237 (Np-237) values were obtained from the alpha energy analyses (AEA) of the plutonium fraction from the ion exchange separation. The Np-239 was spiked into the sample to correct for yields; however, counting room problems and the low yields made it impossible to obtain good Np-239 analyses. Because of the short half-life of Np-239 recounting was not possible. The Np-237 results were corrected using the Pu-236 yields. The reported results for both plutonium and neptunium are so low that it is probably safer to say they are less than 1 to 2 uCi/L (nCi/mL) rather than using the actual values.

The poor yields for the actinide method on 106-AN show the importance of using tracers to correct for separation efficiency. If the actinide levels had been near the TRU limit they would have been reported well below the limit without the use of tracers. Unfortunately there is a serious shortage of Pu-236 tracer available. This could cause problems for waste form qualification analyses where results have to be substantiated for regulatory review. The high variability in Hanford waste make "perfect" procedure development difficult and makes the use of tracers more mandatory. Additional method development is needed to minimize the Pu-236 usage. This development should be directed in several ways:

- o Better knowledge of procedure limitations and corrective actions to provide >90 percent yields.
- o More efficient alpha mounting procedures to reduce Pu-236 concentration requirements for good counting statistics and AEA analyses.
- o More efficient alpha counting instruments to reduce Pu-236 consumption and allow small sample sizes for analysis.

Yesterday's nuclear waste is becoming tomorrow's nuclear product. Characterization and analysis of this product (final waste form) will require the same quality assurance (QA) efforts required for uranium and plutonium products in order to meet the customer's (the public and EPA) specifications. This will require a new level of confidence in waste analyses. No longer will the fastest and cheapest result be the primary goal of the analysis; but the quality and confidence in that value will be of equal or greater importance. This will place additional requirements on analytical methods and operations that will require continued program support.

PART B

PREPARATION OF 106-AN SIMULATED WASTE FOR GROUT FORMULATION TESTING

Plans (References 3, 4, and 5) for formulating and testing grouts with compositions similar to the waste in tank 106-AN called for the development of a nonradioactive simulated waste.

After the problems associated with Na_3PO_4 precipitation in the composite were identified and the initial composite analysis completed, the first 106-AN simulated waste composition was proposed for approval (Reference 6). This initial proposal assumed a 10 to 20 liter batch would be required and that it would be based on the equal volume from each depth composite.

A different formulation was requested (Reference 7) based on evaporator campaign data that estimated the volume fraction of each depth composition. In addition, sodium citrate was to be added to equal the total organic carbon (TOC) concentration. A new simulated waste formulation was calculated (Reference 3) after the major components were analyzed for each sample based on the volume fractions provided. The quantities of carbonate and TOC were based on the equal volume composite results since these analyses were not run on each 106-AN sample. The final formulation information is summarized in Table 2.

In the procedure for preparing the simulated waste, the most important consideration was to be able to prepare and dispense the simulated waste so that a representative (equivalent composition) sample was used for <u>all</u> the planned formulation tests. The best way to do this was to makeup a large single batch of waste rather than several smaller batches. The planned testing program was going to require 20 to 30 liters of synthetic 106-AN. Standards Laboratory had the only large-scale mixer (70 liters) available in the 222S laboratory to make this volume of synthetic.

Originally (Reference 9), we had planned to have Standards Laboratory prepare this material. However, because of absences in their organization and the time frame for completing the project, Analytical Systems Laboratories Unit ended up preparing the synthetic using Standard Laboratories' mixer.

The volume of synthetic prepared was increased to 40 liters since the stirrer operated smoother with less splashing with larger volumes. The mixing tank was calibrated by filling it with 40 liters of water measured using multiple additions with a larger graduated cylinder. The distance from the top of the tank to the top of the liquid was measured to identify to 40 liter level. The tank was flushed with distilled water before preparing the synthetic.

A one liter sample of the synthetic was prepared to identify any dissolution problems that may be encountered. The technical grade sodium aluminate ($NaAlO_2$) used contains a small amount of insoluble materials that makes the solution cloudy. In order to obtain the correct aluminum concentration

with this $NaAlO_2$ the formula that must be used is $NaAl(OH)_4$. The Na_3PO_4 was dissolved before adding to improve its dissolution rate and to simulate the actual precipitation process when its solubility is exceeded. The solid sodium citrate was difficult to dissolve in this test batch. The test batch never became totally clear even at elevated (50 to 60°C) temperature. The solids remaining at elevated temperatures were <5 volume percent and very slow in settling. When the test batch finally cooled and settled for a long time, it contained 15 to 20 percent solids. This is similar to that noted for real 106-AN.

In order to expedite the dissolution of all the 106-AN components and to ensure the maximum homogeneity when transferring the final solution into two 20 liter plastic carboys, it was necessary to heat the larger mixer contents to 30 to 40°C. A large band heater was borrowed from the pilot plant laboratory to do this. All the chemicals were pre-weighed (Table 2) before beginning the preparation. The Na_3PO_4 was dissolved in 7 to 8 liters of water before adding. The sodium citrate was dissolved in 1 to 2 liters of water before adding.

About 15 to 20 liters of distilled water was added to the mixer and the heater turned on for two to three hours before adding the first reagents. Because of the slow temperature rise in the large volume of water, it was decided to add the NaOH first to increase the temperature. In order to control splattering, the reagents were added with the stirrer turned off. After adding the NaOH, the stirrer was turned on and the temperature rose to about 45°C. The reagents were then added in the order and quantity identified in Table 3. A small amount of phosphate solution was spilled when moving this reagent to the mixer. After all the reagents were added and well mixed, water as added to bring the tank to the 40 liter volume as determined by measuring from the top of the tank. The solution was then heated and stirred for about two hours.

After two hours, the solution was transferred by draining the contents into a 20 liter polyethylene carboy while the contents were stirring. A 100 mL sample was taken during the transfer for verification. This procedure was repeated for the second 20 liter carboy of 106-AN synthetic. The analytical results for these samples are shown in Table 2.

The carboys were set up in the grout laboratory with the band heater and a overhead stirrer to ensure homogeneity when sampling (by siphoning) for the grout tests. The 222S laboratory and Analytical Systems Laboratories Unit (ASLU) are only marginally set up to prepare these large volumes of synthetics. In the future, I recommend that the Process Chemistry Laboratories Unit (PCLU) prepare the large volumes of synthetic where the equipment and space are more convenient and provide safer operations.

TABLE 1

106-AN ANALYTICAL RESULTS

Analysis	R2087	R2088	R2089	R2090	Composite R2456
Depth	24' 3"	34' 9"	44' 9"	53'3"	Equal Volume
Visual	Yellow/clear	Yellow/clear	Yellow/clear	Yellow/clear	Yellow/17% Solids
Radiation	50 mR	350 mR	1000 mR	1500 mR	----
Viscosity	1-2 cp	1-2 cp	1cp	<6 cp	----
Density	1.087	1.142	1.344	1.320	1.223 (1.223)
Wt % H_2O	----	----	----	----	66.9%
Na M	1.790	2.500	8.44[RR]	6.90[RR]	5.291[RR] (4.91)
			6.830	6.230	5.434 (4.34)
Al	0.036	0.188	0.771	0.684	0.462 (.420)
K	5.27×10^{-3}	0.017	0.055	0.049	0.039 (.032)
Cr	1.31×10^{-3}	6.14×10^{-3}	0.022	0.019	0.016 (.012)
B	----	5.85×10^{-4}	2.29×10^{-3}	1.98×10^{-3}	1.68×10^{-3}
Ca	2.42×10^{-4}	9.29×10^{-4}	2.93×10^{-3}	2.68×10^{-3}	2.13×10^{-3}
Si	4.76×10^{-4}	6.63×10^{-4}	1.32×10^{-3}	1.55×10^{-3}	1.01×10^{-3}
Fe	----	----	1.71×10^{-4}	----	----
Ni	----	----	9.44×10^{-4}	7.77×10^{-4}	----
Cu	----	8.2×10^{-6}	4.22×10^{-5}	3.27×10^{-5}	2.42×10^{-5}
Mo	----	1.12×10^{-4}	4.93×10^{-4}	4.09×10^{-4}	3.38×10^{-4}
P	0.384	0.175	0.048	0.038	0.202 (.161)
As ug/mL	----	----	----	----	<2.75
Se	----	----	----	----	<0.55
U g/L	----	----	----	----	0.015
Th	----	----	----	----	Incomplete

Table 1 Continued

Analysis	R2087	R2088	R2089	R2090	Composite R2456
OH^- M	0.050	0.346	1.140	1.170	1.353 (0.676)
NO_3^-	0.166	0.583	2.170	2.290	1.452 (1.47)
NO_2^-	0.101	0.353	1.240	1.290	0.799 (0.746)
SO_4^{-2}	3.23×10^{-3}	0.011	0.046	0.048	0.027 (0.027)
PO_4^{-3}	0.373	0.135	0.044	0.043	0.194 (0.149)
Cl^-	9.02×10^{-3}	0.038	0.136	0.140	0.098 (0.081)
F^-	7.34×10^{-3}	$<7.34 \times 10^{-3}$	<0.015	<0.015	1.8×10^{-3}
CN^- (ug/mL)	----	----	----	----	13.8
CO_3^{-2}	----	----	----	----	0.382
TOC	----	----	----	----	0.441
Cs-137	2.62×10^4	1.16×10^5	4.58×10^5	4.65×10^5	2.585×10^5 (2.66×10^5)
Sr-89-90	----	----	----	----	4.36×10^3
Tc-99 (RHO)	----	----	----	----	96.25 (84.7)
Tc-99 (HEDL)	7.97	34.4	147.7	148.6	----
C-14 (HEDL)	0.068	0.25	1.01	1.28	----
H-3	----	----	----	----	6.85
I-129	----	----	----	----	<0.20
Se-79 (HEDL)	0.052	0.23	0.85	0.96	----
TB	----	----	----	----	5.19×10^5
Pu-239/240 uCi/L	----	----	----	----	0.44 ± .16
Pu-238	----	----	----	----	0.21
Am-241	----	----	----	----	1.45
Np-237	----	----	----	----	0.16

( ) - Average value of individual analyses.

RR - Rerun value.

TABLE 2

106-AN SIMULATED WASTE COMPOSITION AND VERIFICATION

Element	Molarity Calculated	Molarity Make-up	Verified M 1st 20L	Verified M 2nd 20L
Na	4.780	5.112	5.14	5.020
Al	0.486	0.486	.488	0.484
OH^-	0.783	0.783	Incomplete	Incomplete
NO_2^-	0.857	0.857	0.834	.828
NO_3^-	1.498	1.498	1.670	1.652
CO_3^{-2}	0.385	0.385	.430	.436
PO_4^{-3}	0.114	0.114	.123 (.125)[a]	0.12 (.124)[a]
SO_4^{-2}	0.031	0.031	.032	0.032
TOC	0.441	0.438	.506	0.505
Cl^-	0.093	0.093	.110	0.107
F^-	0.002	0.002	Incomplete	Incomplete
Ca	0.002	0.002	.00202	0.00193
Cr	0.014	0.014	.0137	.014
K	0.032	0.028	.0288	0.0280

a) Phosphate base on ICP Phosphorus analysis.

TABLE 3

106-AN SIMULATION MAKEUP CALCULATION

Material	Molecular Weight	Molarity	Moles for 40L	Weight for 40L (g)	Actual Weight
NaOH	40.00	0.783	31.32	1252.8	1253.0
$NaAl(OH)_4$	118	0.486	19.44	2293.9	2293.8
$NaNO_3$	84.99	1.498	59.92	5092.6	5092.5
$NaNO_2$	69.00	0.857	34.28	2365.3	2365.8
Na_2CO_3	105.99	0.385	15.40	1632.2	1632.2
Na_2SO_4	142.04	0.031	1.24	176.1	176.1
NaCl	58.44	0.093	3.72	217.4	217.3
NaF	41.99	0.002	0.08	3.36	3.4
$Na_3PO_4 \cdot 12H_2O$	380.12	0.114	4.56	1733.3	1733.0
$Ca(NO_3)_2 \cdot 4H_2O$	236.15	0.002	0.08	18.89	18.8
K_2CrO_4	194.20	0.014	0.56	108.75	108.7
$Na_3Citrate \cdot 2H_2O$	294.10	0.073	2.92	858.90	858.8

8751478
May 8, 1987

R. I. Winters
222S/MO-037/200W
Rockwell Hanford Operations
P. O. Box 800
Richland, WA 99352

^{79}SE AND ^{99}TC MEASUREMENTS IN SAMPLES R-2087 TO R-2090

These samples were analyzed for ^{79}Se and ^{99}Tc using the hydrothermal procedures HTA-4-18 and HTA-4-12 respectively. The samples were acidified prior to the application of the separation procedure.

Attached is a tabular summary of the data. If you have any questions please call A. C. Leaf on 376-3801.

ACLeaf
A. C. Leaf

J. J. McCown, Manager
Chemistry and Analysis

tts

Westinghouse Hanford Company/A subsidiary of Westinghouse Electric Corp / Operating the Hanford Engineering Development Laboratory for the USDOE

ANALYTICAL RESULTS FOR SAMPLES R-2087 TO R-2090

Lab No.	Cust. ID	^{79}Se, d/m/ml	^{99}Tc, d/m/ml
8331	R-2087	1.15×10^2	1.77×10^4
8332	R-2088	5.13×10^2	7.63×10^4
8333	R-2089	1.88×10^3	3.28×10^5
8334	R-2090	2.14×10^3	3.30×10^5

8751377
April 29, 1987

R. I. Winters
222S/MO-037/200W
Rockwell Hanford Operations
P. O. Box 800
Richland, WA 99352

^{14}C MEASUREMENT IN SAMPLES 2087-2090

These samples were analyzed on a Dohrmann TOC Analyzer using the UV-wet oxidation method, with $^{14}CO_2$ trapped in dilute sodium hydroxide. The strong caustic nature of these samples required a modification in the persulfate/phosphoric reagent, resulting in 3.5% persulfate in 2 M phosphoric acid.

The four samples were first tested for any quenching effect on the ^{14}C recovery by introducing an internal standard. A ratio of 50λ standard (traceable to NBS) to 500λ sample was used, with 500λ of this mixture injected. Also direct injection of pure standard ($Na_2\ ^{14}CO_3$) was done to determine ideal recoveries. The results show insignificant quenching effect by NO_x or any other component.

TABLE 1: Standards, Quenching Effects

Sample	Amount	% Recovery
Direct Injection, Standard	500λ	96.5%
Direct Injection, Standard	500λ	98.6%
#2087 (500λ + 50λ Std)	500λ	99.8%
#2088 (500λ + 50λ Std)	500λ	96.5%
#2089 (500λ + 50λ Std)	450λ	98.6%
#2090 (500λ + 50λ Std)	500λ	102.9%

Westinghouse Hanford Company/A subsidiary of Westinghouse Electric Corp / Operating the Hanford Engineering Development Laboratory for the USDOE

The four samples were run in duplicate. The estimated detection limit, under the current conditions is about 0.02 nCi/ml (2×10^{-11} Ci/ml).

TABLE 2: Results

Sample	Volume	cpm	^{14}C (nCi/ml)
#2087	500λ	6	.045
	500λ	12	.091
#2088	500λ	35	0.28
	500λ	29	0.22
#2089	500λ	140	1.06
	500λ	126	0.95
#2090	500λ	166	1.32
	1000λ	312	1.24

In each case the result is corrected for an average 97.6% recovery.

Example calculations (sample 2087a):

$$\frac{6 \text{ cpm} \times 13785 \text{ dpm [std]} \times (15 \text{ ml}/2 \text{ ml dilution})}{12620 \text{ cpm [std]} \times 0.5 \text{ ml} \times 2.22 \times 10^{12} \text{ dpm/Ci} \times .976} = .045$$

If you have any questions please call D. L. Baldwin at 6-3596.

D. L. Baldwin

J. J. McCown, Manager
Chemistry and Analysis

tts

9 2 1 2 4 1 4 0 9 7 7

APPENDIX B

SAMPLING LOCATIONS

TANK 241-AN-106

9 2 1 2 4 1 4 0 9 7 8

This page is intentionally left blank

Westinghouse
Hanford Company

Internal
Memo

WHC-SD-CP-TP-065, Rev. 0
12711-89-028

From: Process Chemistry & Engineering Laboratories
Phone: 3-2779/3-2475 2704S/200W T6-18
Date: March 9, 1989
Subject: 241-AN-106 CHARACTERIZATION AND GROUT FORMULATION HOT CELL TEST PLAN

To: W. G. Richmond R1-48

cc:	R. E. Brandt	T6-30	T. A. Lane	T6-12
	J. D. Briggs	T6-14	J. R. Prilucik	T6-26
	S. A. Catlow	T6-30	D. A. Reynolds	R2-11
	R. D. Claghorn	R1-48	D. E. Scully	R1-51
	D. A. Dodd	T6-50	C. M. Seidel	T6-50
	R. K. Fuller	T6-30	J. P. Sloughter	T6-18
	A. P. Hammitt	T6-50	L. H. Taylor	T6-16
	T. G. Ibsen	T6-16	W. I. Winters	T6-50
	S. A. Jones	T6-30	TLW-LJ File/LB	

References: (1) WHC-EP-0182-9, "Tank Farm Surveillance and Waste Status Summary Report for December 1988," released January 1989, J. M. Thurman.

(2) SD-WM-TP-061, Rev. 0, "241-AN-106 Characterization and Grout Formulation Hot Cell Test Plan," released January 20, 1989, A. P. Hammitt.

(3) Internal Letter #65452-87-198, W. I. Winters to W. G. Richmond, "106-AN Analytical Results and Simulate Development," July 21,1987.

OBJECTIVE

The objective of this letter is to describe the statistical methods and controls to be used in the Tank 106-AN characterization test plan. The purpose of the 241-AN-106 characterization and grout formulation hot cell test plan is:

- to characterize Tank 106-AN waste for chemical and radiological components
- to determine the grout formulation and grout product properties
- to quantify analytical methods and develop equipment for use in the hot cell.

SAMPLING

Samples (sample size 100 ml) from Tank 106-AN will be obtained based on a random sampling plan. Current data (see Reference 3) suggest that

Hanford Operations and Engineering Contractor for the US Department of Energy

54-7600-081

Tank 106-AN is stratified but the interfaces are not defined. The following random sampling plan will be used to obtain samples which will represent the entire tank without sampling one strata more than another.

An elevation was randomly selected from each horizontal section beneath each of the three risers identified for sampling. Elevation is the height in inches from the bottom of the tank. The horizontal sections were defined by dividing the liquid height of the tank by four. The liquid height of Tank 106-AN is 377.5 inches as stated in Reference 1.

Using the program SAMPLE of the statistical package MSUSTAT, which uses a random number generator, three elevations for each horizontal section were selected. For Tank 106-AN the four randomly selected elevations for each riser are

Riser	Elevations			
22A	20	100	220	331
1B	49	137	224	292
16C	16	114	210	366

The three risers were also randomly selected from the ten available risers using the random number generator from the program SAMPLE of MSUSTAT.

ANALYTICAL MEASUREMENTS

A total of twelve sample bottles from Tank 106-AN will be received in the 1F hot cell of the 222-S Analytical Laboratory. The contents of each sample bottle will be analyzed in duplicate following the procedure listed in Reference 2 for sample preparation.

The twenty-four samples will be analyzed for the specific analytes listed in Attachment 1. A standard(s) for each analytical procedure shall be analyzed with each batch of samples. The percent recovery value(s) shall be reported with each sample result.

If the agreement between duplicate analyses for each analyte measured is not within the limits explained in Attachment 2, then two new aliquots will be taken from the original sample, prepared, and resubmitted for analytical measurement. The resulting values will be statistically evaluated prior to use in final calculations.

COMPOSITE SAMPLE

A composite sample will be made based on the analytical results from the individual samples. Analysis of the analytical data will use a

statistical method known as analysis of variance (ANOVA) to test three hypotheses for statistically significant differences in concentration.

- differences within a section
- differences between the sections
- differences between the risers

The above three sources of variability are explained in Attachment 2. The analytical error, used as the reference value in the above tests, is estimated from the differences between the replicate analyses.

If the statistical analyses of the data indicate stratification rather than homogeneity of the tank contents, then a composite sample formulation will be developed to agree with the statistical test results. Otherwise, the composite will be made by combining equal parts by volume from each of the twelve individual samples.

The composite sample will be analyzed in duplicate for the specific analytes listed in Attachment 1. The procedures for sample preparation are listed in Reference 2.

If the TOC analytical result is greater than 2 g/l then a portion of the composite will be submitted to the 300 Area Analytical Laboratory for organic analyses. The specific organic analyses requested are listed in Reference 2.

The agreement between the duplicate analyses must be within the limits as specified in Attachment 2. If the agreement is outside the limits, then two new aliquots of the composite sample will be taken, prepared, and resubmitted for analytical measurement.

CHARACTERIZATION

It is requested that all analytical laboratory results (original and reruns) be reported to A. P. Hammitt. Please include with the raw data any notation of why data may be suspect. All analyses, both samples and standards, are to be reported on the form given as Attachment 3.

The analytical results from the twelve individual samples will be compared to the analytical results from the composite for each component measured using ANOVA. This comparison will assist in determining the applicability of using the composite sample data instead of the individual sample data to define the contents of the tank.

The mean of the analytical results from the composite sample for each component measured will be calculated and reported as the concentration of Tank 106-AN.

GROUT SAMPLES

The grout mixtures will be made using aliquots from the composite sample solution after the characterization of the composite solution is completed; i.e., all analyses have been reported by the analytical laboratory and the Statistics Team have analyzed the results. Three grout mixtures will be made for each of the following measurements:

- EP Toxicity
- compressive strength
- ANS 16.1 Leach Indices
- drainable liquid

In addition, viscosity, gel strength, and density will also be measured for each grout mixture.

If you have any questions, please call L. Jensen (373-2779), T. L. Welsh (373-2475), or A. P. Hammitt (373-4203).

T L Welsh

T. L. Welsh
Statistician

L. Jensen
Statistician

mcr

Attachments 3

ATTACHMENT 1

I=Individual Sample
C=Composite Sample

Chemical	Method	Procedure	Quality Control Index Number	Sample
Ag	ICP	LA-505-143	S047	I,C
Al	ICP	LA-505-143	S013	I,C
Ba	ICP	LA-505-143	S056	I,C
Bi	ICP	LA-505-143	S083	I,C
Ca	ICP	LA-505-143	S020	I,C
Cd	ICP	LA-505-143	S048	I,C
Cr	ICP	LA-505-143	S024	I,C
Cu	ICP	LA-505-143	S029	I,C
Fe	ICP	LA-505-143	S026	I,C
K	ICP	LA-505-143	S019	I,C
Mg	ICP	LA-505-143	S012	I,C
Mn	ICP	LA-505-143	S025	I,C
Mo	ICP	LA-505-143	S042	I,C
Na	ICP	LA-505-143	S011	I,C
Pb	ICP	LA-505-143	S082	I,C
P	ICP	LA-505-143	S015	I,C
Si	ICP	LA-505-143	S014	I,C
Ti	ICP	LA-505-143	S022	I,C
Zn	ICP	LA-505-143	S030	I,C
Zr	ICP	LA-505-143	S040	I,C

ATTACHMENT 1
(continued)

I=Individual Sample
C=Composite Sample

Chemical	Method	Procedure	Quality Control Index Number	Sample
Cl	Dionex	LA-533-105	R972	I,C
F	Dionex	LA-533-105	R974	I,C
NO_3	Dionex	LA-533-105	R978	I,C
NO_2	Dionex	LA-533-105	----	I,C
	Spec	LA-645-001	R760	I,C
PO_4	Dionex	LA-533-105	R976	I,C
SO_4	Dionex	LA-533-105	R970	I,C
Density	Density	LA-510-112	R820	I,C
Co-60	GEA	LA-548-121	R905	I,C
Ru-Rh-106	GEA	LA-548-121	----	I,C
Cs-137	GEA	LA-548-121	R901	I,C
Nb-94	GEA	LA-548-121	No Std	C
Cs-134	GEA	LA-548-121	----	C
As	AAS	LA-355-131	R741	C
Hg	AAS	LA-325-102	----	C
Se	AAS	LA-365-131	R743	C
Am-241	sep/AEA	LA-503-156	R201	C
CN	dist/spec	*		C
CO_3	TOC	LA-622-101	R788	C
TOC	TOC	LA-344-101	R784	C
% H_2O	Evap	LA-564-101	R824	C
OH	Titrat	LA-661-102	R800	C

ATTACHMENT 1
(continued)

I=Individual Sample
C=Composite Sample

Chemical	Method	Procedure	Quality Control Index Number	Sample
U	Fluor	LA-925-106	R797	C
H-3	sep/LSC	LA-218-111	E348	C
C-14	sep/LSC	LA-348-103	S908	C
Tc-99	sep/LSC	LA-438-101	R938	C
Se-79	sep/LSC	**		C
Sr-90	sep/Beta	LA-220-101	R916	C
I-129	sep/LE GEA	LA-378-103	R928	C
Np-237	sep/Alpha/AEA	LA-933-141	R932	C
Pu-238	sep/Alpha/AEA	LA-503-156	No Std	C
Pu-239/240	sep/Alpha/AEA	LA-503-156	R211	C
Cm-244	sep/Alpha/AEA	LA-503-156	No Std	C

ICP = Inductively Coupled Plasma Spectrophotometer
Dionex = Ion Chromatography
AAS = Atomic Absorption Spectrophotometer
Spec = Spectrophotometer
TOC = Total Organic Carbon
Evap = Evaporation and Weighing
Titrat = Autotitrator
Fluor = Laser Fluorimeter
GEA = GAMMA Energy Analysis
dist/spec = distillation along with spectrophotometer
sep/LSC = chemical separation along with Liquid Scintillation Counting
sep/Beta = chemical separation along with Total Beta Proportional Counting
sep/LE GEA = chemical separation along with Low Energy GAMMA Energy Analysis
sep/Alpha/AEA = chemical separation with Alpha Proportional Counting and Alpha Energy Analysis

* ASL will determine using SD-WM-TI-315 or PNL will determine using modified SW-846

** PNL will determine using HTA-4-18

This page is intentionally left blank

ATTACHMENT 2

The agreement between the duplicates for each pair of sample analytical results by analyte will be evaluated by calculating the absolute percent difference. If A and B represent the duplicate analytical results then the absolute percent difference is equal to the absolute value of (A-B) divided by the mean value of A and B multiplied by 100. The agreement between duplicate analyses shall be within 3 sigma where sigma is defined to be the standard deviation of the standards analyzed by each measurement method. The standards and the associated summary statistics are reported by the Laboratory Measurements and Control System (LMCS). If the agreement is outside the 3 sigma limit, then two new dilutions will be made from the original sample and submitted for analysis.

The comparison of the individual sample analytical results for within a section, between sections, and between risers will be evaluated at the 0.05 level of significance using a statistical procedure called analysis of variance (ANOVA). Both the within and between section comparisons will indicate whether or not the tank contents are stratified. The between risers comparison will provide an "overall" indication of heterogeneity within the tank contents. The comparisons will also be evaluated by using nonparametric statistical tests.

If an analytical result is initially reported as a less than value, then the chemist in charge of the analytical method will be consulted. The possible outcomes of the consultation are:

- rerun the samples with a different dilution
- rerun the samples with a longer count time
- accept the less than value (at or below the detection limits).

This page is intentionally left blank

ATTACHMENT 3

Sample Number_____ Date Submitted_____ Date Analyzed_____

Analyte	Analytical Result	Standard Result(s)
Al		
Ba		
Bi		
Cd		
Ca		
Cr		
Cu		
Fe		
Pb		
Mg		
Mn		
Mo		
P		
K		
Si		
Ag		
Na		
Ti		
Zn		
Zr		

ATTACHMENT 3
(continued)

Analyte	Analytical Result	Standard Result(s)	Date Submitted	Date Analyzed
F				
Cl				
NO_2 (Dionex)				
NO_3				
PO_4				
SO_4				
NO_2 (Spec)				
Co-60				
Cs-137				
Cs-134				
Nb-94				
Ru-Rh-106				
Density				

In addition the following analyses are required for the composite samples

As				
Hg				
Se				
CO_3				
TOC				
H_2O				
OH				
U				

9 2 1 2 4 1 0 9 9 0

ATTACHMENT 3
(continued)

Analyte	Analytical Result	Standard Result(s)	Date Submitted	Date Analyzed
CN				
H-3				
C-14				
Se-79				
Sr-90				
Tc-99				
I-129				
Am-241				
Np-237				
Pu-238				
Pu-239/240				
Cm-244				

This page is intentionally left blank

9 2 1 2 4 1 4 0 9 9 3

APPENDIX C

INDIVIDUAL SAMPLE RESULTS

TANK 241-AN-106

9 2 1 2 4 1 4 0 9 9 4

This page is intentionally left blank

The analytical results for the twenty-four subsamples are located in the following tables. The two sub-samples for each sample number are designated in the tables as "a" and "b"; e.g. the two subsamples for sample number 1 are designated as 1a and 1b.

The mean is calculated by adding the analytical results for the "a" and "b" subsamples and dividing by two. If A and B represent the analytical results for the "a" and "b" subsamples, then the percent difference (%Diff.) is calculated by dividing the absolute value of the difference between A and B by the mean of A and B and then multiplying by 100.

$$\%\text{Diff.} = \frac{|A - B|}{(A + B) / 2} * 100$$

This page is intentionally left blank

9 2 1 2 4 1 4 0 9 9 6

Constituent	Sample Number / Units	1a	1b	Mean	%Diff.
Aluminum	mg/L	1452	1463	1458	0.8%
Barium	mg/L	<[1] 6.072	< 6.072	< 6.072	
Bismuth	mg/L	< 79.2	< 79.2	< 79.2	
Cadmium	mg/L	< 27.06	< 27.06	< 27.06	
Calcium	mg/L	10.703	10.351	10.527	3.3%
Chromium	mg/L	105.71	105.27	105.49	0.4%
Copper	mg/L	< 2.046	< 2.046	< 2.046	
Iron	mg/L	< 3.762	< 3.762	< 3.762	
Lead	mg/L	< 250.8	< 250.8	< 250.8	
Magnesium	mg/L	< 1.122	< 1.122	< 1.122	
Manganese	mg/L	< 30.36	< 30.36	< 30.36	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 36.3	< 36.3	< 36.3	
Phosphorus	mg/L	10637	9889	10263	7.3%
Potassium	mg/L	293.7	294.8	294.3	0.4%
Silver	mg/L	< 1.98	< 1.98	< 1.98	
Sodium	mg/L	38390	40590	39490	5.6%
Titanium	mg/L	< 1.914	< 1.914	< 1.914	
Zinc	mg/L	< 5.148	< 5.148	< 5.148	
Zirconium	mg/L	< 15.18	< 15.18	< 15.18	
Carbonate	M	0.139		0.139	
Chloride	mg/L	1089	1232	1161	12.3%
Hydroxide	M	0.0866		0.0866	
Fluoride	mg/L	< 11.0	< 147.4	< 79.2	
Nitrite	M	0.1408	0.1441	0.1425	2.3%
Nitrate	mg/L	18260	17600	17930	3.7%

[1] < indicates a less than value

9 2 1 2 4 1 4 0 9 9 7

Constituent	Sample Number / Units	1a	1b	Mean	%Diff.
Phosphate	mg/L	29480	29810	29645	1.1%
Sulfate	mg/L	2860	2772	2816	3.1%
Cobalt-60	µCi/L	< 3.806	14.52	9.16	116.9%
Niobium-94	µCi/L	< 7.568	< 5.456	< 6.512	
RuRh-106	µCi/L	< 533.5	< 233.2	< 383.4	
Cesium-134	µCi/L	< 8.239	< 5.06	< 6.65	
Cesium-137	µCi/L	42350	43450	42900	2.6%
Density	g/cm^3	1.11	1.10	1.11	0.9%

9 2 1 2 4 1 4 0 9 9 8

Constituent	Sample Number / Units	2a	2b	Mean	%Diff.
Aluminum	mg/L	1485	1441	1463	3.0%
Barium	mg/L	< 6.072	< 6.072	< 6.072	
Bismuth	mg/L	< 79.2	< 79.2	< 79.2	
Cadmium	mg/L	< 27.06	< 27.06	< 27.06	
Calcium	mg/L	12.21	10.406	11.31	16.0%
Chromium	mg/L	103.18	104.61	103.90	1.4%
Copper	mg/L	< 2.046	< 2.046	< 2.046	
Iron	mg/L	< 3.762	< 41.36	< 22.56	
Lead	mg/L	< 250.8	< 250.8	< 250.8	
Magnesium	mg/L	< 1.122	< 1.122	< 1.122	
Manganese	mg/L	< 30.36	< 30.36	< 30.36	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 36.3	< 66.55	< 51.4	
Phosphorus	mg/L	9779	9658	9719	1.2%
Potassium	mg/L	343.2	302.5	322.9	12.6%
Silver	mg/L	< 1.98	< 3.63	< 2.81	
Sodium	mg/L	41140	40260	40700	2.2%
Titanium	mg/L	< 1.914	< 3.509	< 2.712	
Zinc	mg/L	< 5.148	< 9.438	< 7.293	
Zirconium	mg/L	< 15.18	< 27.83	< 21.51	
Carbonate	M	0.136		0.136	
Chloride	mg/L	946	1166	1056	20.8%
Hydroxide	M	0.091		0.091	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	0.143	0.1375	0.140	3.9%
Nitrate	mg/L	18260	18370	18315	0.6%
Phosphate	mg/L	29370	29590	29480	0.7%

9 3 1 2 4 1 4 0 9 9 9

Constituent	Sample Number / Units	2a	2b	Mean	%Diff.
Sulfate	mg/L	2750	2673	2712	2.8%
Cobalt-60	µCi/L	< 20.24	< 5.17	< 12.71	
Niobium-94	µCi/L	< 17.82	< 7.777	< 12.80	
RuRh-106	µCi/L	< 1320	< 476.3	< 898	
Cesium-134	µCi/L	< 26.18	< 6.864	< 16.52	
Cesium-137	µCi/L	46310	41910	44110	10.0%
Density	g/cm^3	1.11	1.11	1.11	0.0%

9 2 1 2 4 1 1 0 0 0

Constituent	Sample Number / Units	3a	3b	Mean	%Diff.
Aluminum	mg/L	19910	21450	20680	7.4%
Barium	mg/L	< 14.52	< 21.23	< 17.875	
Bismuth	mg/L	< 189.2	< 277.2	< 233.2	
Cadmium	mg/L	< 64.46	< 94.71	< 79.585	
Calcium	mg/L	166.1	154	160.1	7.6%
Chromium	mg/L	1155	1144	1150	1.0%
Copper	mg/L	< 4.873	< 7.161	< 6.017	
Iron	mg/L	< 8.965	< 13.2	< 11.0825	
Lead	mg/L	< 597.3	< 877.8	< 737.6	
Magnesium	mg/L	< 2.673	< 3.927	< 3.300	
Manganese	mg/L	< 72.38	< 106.26	< 89.32	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 86.46	< 127.6	< 107.0	
Phosphorus	mg/L	1452	1364	1408	6.3%
Potassium	mg/L	2101	1925	2013	8.7%
Silver	mg/L	< 4.719	< 6.93	< 5.82	
Sodium	mg/L	162800	167200	165000	2.7%
Titanium	mg/L	< 4.565	< 6.699	< 5.632	
Zinc	mg/L	< 12.32	< 18.04	< 15.18	
Zirconium	mg/L	< 36.19	< 53.13	< 44.66	
Carbonate	M	0.607		0.607	
Chloride	mg/L	#[2] 53570	# 38390	5429	3.9%
Chloride RR	mg/L	5533	5324		
Hydroxide	M	1.11		1.11	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	

[2] # indicates outlier replaced by rerun value.

Constituent	Sample Number / Units	3a	3b	Mean	%Diff.
Nitrite	M	1.232	1.221	1.2265	0.9%
Nitrate	mg/L	161700	161700	161700	0.0%
Phosphate	mg/L	# 1067	# 1009.8	3454	26.8%
Phosphate RR	mg/L	2992	3916		
Sulfate	mg/L	# 234300	# 232100	4433	3.0%
Sulfate RR	mg/L	4367	4499		
Cobalt-60	µCi/L	< 35.2	< 57.09	< 46.1	
Niobium-94	µCi/L	< 72.93	< 75.35	< 74.14	
RuRh-106	µCi/L	< 5401	< 5467	< 5434	
Cesium-134	µCi/L	< 75.9	< 72.49	< 74.2	
Cesium-137	µCi/L	434500	444400	439450	2.3%
Cesium-137 RR	µCi/L	*[3] 438900	* 443300		
Density	g/cm^3	1.37	1.36	1.37	0.7%

[3] * indicates rerun value not used as it is statistically the same as the original value.

9 2 1 2 4 1 1 0 0 2

Constituent	Sample Number / Units	4a	4b	Mean	%Diff.
Aluminum	mg/L	18700	20790	19745	10.6%
Barium	mg/L	< 14.52	< 21.23	< 17.88	
Bismuth	mg/L	< 189.2	< 277.2	< 233.2	
Cadmium	mg/L	< 64.46	< 94.71	< 79.59	
Calcium	mg/L	145.2	165	155	12.8%
Chromium	mg/L	1133	1177	1155	3.8%
Copper	mg/L	5.203	< 7.161	6.182	31.7%
Iron	mg/L	< 8.965	< 13.2	< 11.1	
Lead	mg/L	< 597.3	< 877.8	< 737.6	
Magnesium	mg/L	< 2.673	< 3.927	< 3.300	
Manganese	mg/L	< 72.38	< 106.26	< 89.32	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 86.46	< 127.6	< 107.0	
Phosphorus	mg/L	4741	4664	4703	1.6%
Potassium	mg/L	2002	2024	2013	1.1%
Silver	mg/L	< 4.719	< 6.93	< 5.82	
Sodium	mg/L	158400	169400	163900	6.7%
Titanium	mg/L	< 4.565	< 6.699	< 5.632	
Zinc	mg/L	< 12.32	< 18.04	< 15.18	
Zirconium	mg/L	< 36.19	< 53.13	< 44.66	
Carbonate	M	0.618		0.618	
Chloride	mg/L	# 59730	# 39050	4015	12.1%
Chloride RR	mg/L	3773	4257		
Hydroxide	M	0.782		0.782	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	1.166	1.199	1.183	2.8%
Nitrate	mg/L	156200	152900	154550	2.1%

9 2 1 2 4 1 1 0 0 3

Constituent	Sample Number / Units	4a	4b	Mean	%Diff.
Phosphate	mg/L	4180	4466	4323	6.6%
Sulfate	mg/L	# 239800	# 227700	3652	5.4%
Sulfate RR	mg/L	3553	3751		
Cobalt-60	µCi/L	< 16.06	< 60.5	< 38.3	
Niobium-94	µCi/L	< 62.26	< 61.71	< 61.99	
RuRh-106	µCi/L	< 3718	< 4752	< 4235	
Cesium-134	µCi/L	< 67.54	< 74.69	< 71.12	
Cesium-137	µCi/L	415800	443300	429550	6.4%
Cesium-137 RR	µCi/L	* 327800	* 342100		
Density	g/cm^3	1.65	1.54	1.60	6.9%

9 2 1 2 4 1 4 1 0 0 4

Constituent	Sample Number / Units	5a	5b	Mean	%Diff.
Aluminum	mg/L	1495.8	1461.2	1479	2.3%
Barium	mg/L	< 6.04	< 6.13	< 6.09	
Bismuth	mg/L	< 79.31	< 79.31	< 79.31	
Cadmium	mg/L	< 27.08	< 27.08	< 27.08	
Calcium	mg/L	11.242	10.93	11.09	2.8%
Chromium	mg/L	106.39	104.68	105.54	1.6%
Copper	mg/L	< 2.027	< 2.027	< 2.027	
Iron	mg/L	< 3.75	< 3.75	< 3.75	
Lead	mg/L	< 250.7	< 250.7	< 250.7	
Magnesium	mg/L	< 1.123	< 1.123	< 1.123	
Manganese	mg/L	< 30.34	< 30.34	< 30.34	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 36.3	< 36.3	< 36.3	
Phosphorus	mg/L	10357.6	9471.85	9914.7	8.9%
Potassium	mg/L	308.4	276.1	292.3	11.1%
Silver	mg/L	< 2.017	< 2.017	< 2.017	
Sodium	mg/L	41979	38439	40209	8.8%
Titanium	mg/L	< 1.90	< 1.90	< 1.90	
Zinc	mg/L	< 5.18	< 5.18	< 5.18	
Zirconium	mg/L	< 15.15	< 15.15	< 15.15	
Carbonate	M	0.148		0.148	
Chloride	mg/L	605	585.2	595	3.3%
Hydroxide	M	0.0888		0.0888	
Fluoride	mg/L	< 139.39	< 111.1	< 125.2	
Nitrite	M	0.1024	0.0999	0.1012	2.5%
Nitrate	mg/L	17050	15840	16445	7.4%
Phosphate	mg/L	30800	29480	30140	4.4%

9 2 1 2 4 1 4 1 0 0 5

Constituent	Sample Number / Units	5a	5b	Mean	%Diff.
Sulfate	mg/L	< 1485	499.4	499	
Cobalt-60	µCi/L	< 2.86	< 1.694	< 2.28	
Niobium-94	µCi/L	< 11.99	< 9.273	< 10.63	
RuRh-106	µCi/L	< 468.6	< 420.2	< 444.4	
Cesium-134	µCi/L	< 11.99	< 8.833	< 10.41	
Cesium-137	µCi/L	41910	42570	42240	1.6%
Density	g/cm^3	1.11	1.12	1.12	0.9%

9 2 1 2 4 1 4 1 0 0 6

Constituent	Sample Number / Units	6a	6b	Mean	%Diff.
Aluminum	mg/L	2582	2457	2520	5.0%
Barium	mg/L	< 6.04	< 6.04	< 6.04	
Bismuth	mg/L	< 79.31	< 79.31	< 79.31	
Cadmium	mg/L	< 27.08	< 27.08	< 27.08	
Calcium	mg/L	18.87	16.27	17.57	14.8%
Chromium	mg/L	169.9	167.6	168.8	1.4%
Copper	mg/L	< 2.027	< 2.027	< 2.027	
Iron	mg/L	< 3.75	< 3.75	< 3.75	
Lead	mg/L	< 250.7	< 250.7	< 250.7	
Magnesium	mg/L	< 1.123	< 1.123	< 1.123	
Manganese	mg/L	< 30.34	< 30.34	< 30.34	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 36.3	< 36.3	< 36.3	
Phosphorus	mg/L	8347.5	7768	8058	7.2%
Potassium	mg/L	424.1	377.2	400.7	11.7%
Silver	mg/L	< 2.017	< 2.017	< 2.017	
Sodium	mg/L	44003	41474	42739	5.9%
Titanium	mg/L	< 1.90	< 1.90	< 1.90	
Zinc	mg/L	< 5.18	< 5.18	< 5.18	
Zirconium	mg/L	< 15.15	< 15.15	< 15.15	
Carbonate	M	0.176		0.176	
Chloride	mg/L	900.9	976.8	938.9	8.1%
Hydroxide	M	0.142		0.142	
Fluoride	mg/L	< 139.39	< 111.1	< 125.2	
Nitrite	M	0.1782	0.1859	0.1821	4.2%
Nitrate	mg/L	22913	21780	22347	5.1%
Phosphate	mg/L	25960	24090	25025	7.5%

9 2 1 2 4 1 4 1 0 0 7

Constituent	Sample Number / Units	6a	6b	Mean	%Diff.
Sulfate	mg/L	< 1485	726	840	
Sulfate RR	mg/L		{[4] 953.7		
Cobalt-60	µCi/L	<@[5] 476.3	12.32	12.32	
Niobium-94	µCi/L	< 10.131	< 7.612	< 8.872	28.4%
RuRh-106	µCi/L	< 646.8	< 394.9	< 520.9	48.4%
Cesium-134	µCi/L	< 11	< 7.7	< 9	35.3%
Cesium-137	µCi/L	63580	63250	63415	0.5%
Density	g/cm^3	1.13	1.12	1.13	0.9%

[4] { indicates original and rerun value averaged.

[5] @ indicates outlier not rerun.

9 2 1 2 4 1 4 1 0 0 8

Constituent	Sample Number / Units	7a	7b	Mean	%Diff.
Aluminum	mg/L	1391.9	1445.3	1418.6	3.8%
Barium	mg/L	< 6.04	< 6.04	< 6.04	
Bismuth	mg/L	< 79.31	< 79.31	< 79.31	
Cadmium	mg/L	< 27.08	< 27.08	< 27.08	
Calcium	mg/L	10.23	9.79	10.01	4.4%
Chromium	mg/L	99.53	102.96	101.25	3.4%
Copper	mg/L	< 2.027	< 2.027	< 2.027	
Iron	mg/L	< 3.75	< 3.75	< 3.75	
Lead	mg/L	< 250.7	< 250.7	< 250.7	
Magnesium	mg/L	< 1.123	< 1.123	< 1.123	
Manganese	mg/L	< 30.34	< 30.34	< 30.34	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 36.3	< 36.3	< 36.3	
Phosphorus	mg/L	9540	8756	9148	8.6%
Potassium	mg/L	333.8	283.0	308.4	16.5%
Silver	mg/L	< 2.017	< 2.017	< 2.017	
Sodium	mg/L	38186	36163	37175	5.4%
Titanium	mg/L	< 1.90	< 1.90	< 1.90	
Zinc	mg/L	< 5.18	< 5.18	< 5.18	
Zirconium	mg/L	< 15.15	< 15.15	< 15.15	
Carbonate	M	0.134		0.134	
Chloride	mg/L	686.4	683.1	684.8	0.5%
Hydroxide	M	0.072		0.072	
Fluoride	mg/L	< 111.1	< 111.1	< 111.1	
Nitrite	M	0.0846	0.11	0.10	26.1%
Nitrate	mg/L	15180	14850	15015	2.2%
Phosphate	mg/L	27940	27060	27500	3.2%

9 2 1 2 4 1 1 0 0 9

Constituent	Sample Number / Units	7a	7b	Mean	%Diff.
Sulfate	mg/L	530.2	537.9	534.1	1.4%
Cobalt-60	µCi/L	< 3.641	< 7.623	< 5.632	
Niobium-94	µCi/L	< 12.43	< 8.437	< 10.43	
RuRh-106	µCi/L	< 658.9	< 749.1	< 704.0	
Cesium-134	µCi/L	< 12.76	< 10.142	< 11.45	
Cesium-137	µCi/L	41910	40920	41415	2.4%
Density	g/cm^3	1.10	1.08	1.09	1.8%

9 2 1 2 4 1 4 1 0 1 0

Constituent	Sample Number / Units	8a	8b	Mean	%Diff.
Aluminum	mg/L	20020	20680	20350	3.2%
Barium	mg/L	< 14.52	< 21.23	< 17.88	
Bismuth	mg/L	< 189.2	< 277.2	< 233.2	
Cadmium	mg/L	< 64.46	< 94.71	< 79.59	
Calcium	mg/L	132	158.4	145	18.2%
Chromium	mg/L	1144	1188	1166	3.8%
Copper	mg/L	< 4.873	< 7.161	< 6.017	
Iron	mg/L	< 8.965	< 13.2	< 11.1	
Lead	mg/L	< 597.3	< 877.8	< 737.6	
Magnesium	mg/L	< 2.673	< 3.927	< 3.300	
Manganese	mg/L	< 72.38	< 106.26	< 89.32	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 86.46	< 127.6	< 107.0	
Phosphorus	mg/L	1298	1419	1359	8.9%
Potassium	mg/L	1848	2035	1942	9.6%
Silver	mg/L	< 4.719	< 6.93	< 5.82	
Sodium	mg/L	143000	167200	155100	15.6%
Titanium	mg/L	< 4.565	< 6.699	< 5.632	
Zinc	mg/L	< 12.32	< 18.04	< 15.18	
Zirconium	mg/L	< 36.19	< 53.13	< 44.66	
Carbonate	M	0.551		0.551	
Chloride	mg/L	4642	4752	4697	2.3%
Hydroxide	M	1.12		1.12	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	1.276	1.243	1.260	2.6%
Nitrate	mg/L	158400	173800	166100	9.3%
Phosphate	mg/L	3773	3806	3790	0.9%

9 2 1 2 4 1 4 1 0 1 1

Constituent	Sample Number / Units	8a	8b	Mean	%Diff.
Sulfate	mg/L	4565	4576	4571	0.2%
Cobalt-60	µCi/L	< 23.1	< 51.7	< 37.4	
Niobium-94	µCi/L	< 54.34	< 66.33	< 60.34	
RuRh-106	µCi/L	< 3333	< 4906	< 4120	
Cesium-134	µCi/L	< 56.43	< 70.73	< 63.58	
Cesium-137	µCi/L	444400	446600	445500	0.5%
Density	g/cm^3	1.36	1.35	1.36	0.7%

9 2 1 2 4 1 1 1 0 1 2

Constituent	Sample Number / Units	9a	9b	Mean	%Diff.
Aluminum	mg/L	1364	1463	1414	7.0%
Barium	mg/L	< 6.072	< 6.072	< 6.072	
Bismuth	mg/L	< 79.2	< 79.2	< 79.2	
Cadmium	mg/L	< 27.06	< 27.06	< 27.06	
Calcium	mg/L	{ 10.01	{ 54.01	24.08	58.4%
Calcium RR		24.09	8.217		
Chromium	mg/L	105.82	105.82	105.82	0.0%
Copper	mg/L	< 2.046	< 2.046	< 2.046	
Iron	mg/L	< 3.762	< 3.762	< 3.762	
Lead	mg/L	< 250.8	< 250.8	< 250.8	
Magnesium	mg/L	< 1.122	4.301	2.712	117.2%
Manganese	mg/L	< 30.36	< 30.36	< 30.36	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 36.3	< 36.3	< 36.3	
Phosphorus	mg/L	9185	10010	9598	8.6%
Potassium	mg/L	280.5	294.8	287.7	5.0%
Silver	mg/L	< 1.98	< 1.98	< 1.98	
Sodium	mg/L	36080	40700	38390	12.0%
Titanium	mg/L	< 1.914	< 1.914	< 1.914	
Zinc	mg/L	< 5.148	< 5.148	< 5.148	
Zirconium	mg/L	< 15.18	< 14.3	< 14.7	
Carbonate	M	0.178		0.178	
Chloride	mg/L	642.4	649	646	1.0%
Hydroxide	M	0.245		0.245	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	0.1441	0.1386	0.1414	3.9%
Nitrate	mg/L	16170	16280	16225	0.7%

9 2 1 2 4 1 4 1 0 1 3

Constituent	Sample Number / Units	9a	9b	Mean	%Diff.
Phosphate	mg/L	29040	29480	29260	1.5%
Sulfate	mg/L	886.6	899.8	893.2	1.5%
Cobalt-60	µCi/L	< 4.543	10.978	7.761	82.9%
Niobium-94	µCi/L	< 6.908	< 5.896	< 6.402	
RuRh-106	µCi/L	< 523.6	< 338.8	< 431.2	
Cesium-134	µCi/L	< 7.645	< 5.775	< 6.710	
Cesium-137	µCi/L	42240	43560	42900	3.1%
Density	g/cm^3	1.11	1.12	1.12	0.9%

9 2 1 2 4 1 4 1 0 1 4

Constituent	Sample Number / Units	10a	10b	Mean	%Diff.
Aluminum	mg/L	12430	15070	13750	19.2%
Barium	mg/L	< 11.11	< 14.52	< 12.82	
Bismuth	mg/L	< 145.2	< 189.2	< 167.2	
Cadmium	mg/L	< 49.61	< 64.46	< 57.04	
Calcium	mg/L	122.1	125.4	123.8	2.7%
Chromium	mg/L	772.2	888.8	830.5	14.0%
Copper	mg/L	< 3.751	< 4.873	< 4.312	
Iron	mg/L	< 6.897	< 8.965	< 7.931	
Lead	mg/L	< 459.8	< 597.3	< 528.6	
Magnesium	mg/L	< 2.057	< 2.673	< 2.365	
Manganese	mg/L	< 55.66	< 72.38	< 64.02	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 66.55	< 86.46	< 76.51	
Phosphorus	mg/L	3432	3927	3680	13.5%
Potassium	mg/L	1188	1309	1249	9.7%
Silver	mg/L	< 3.63	< 4.719	< 4.17	
Sodium	mg/L	90640	116600	103620	25.1%
Titanium	mg/L	< 3.509	< 4.565	< 4.037	
Zinc	mg/L	< 9.438	< 12.32	< 10.88	
Zirconium	mg/L	< 27.83	< 36.19	< 32.01	
Carbonate	M	0.443		0.443	
Chloride	mg/L	3234	3344	3289	3.3%
Hydroxide	M	0.508		0.508	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	0.8261	0.8481	0.8371	2.6%
Nitrate	mg/L	94688	# 223300	95805	2.3%
Nitrate RR	mg/L		96921		

Constituent	Sample Number / Units	10a	10b	Mean	%Diff.
Phosphate	mg/L	10637	10923	10780	2.7%
Sulfate	mg/L	3102	3146	3124	1.4%
Cobalt-60	µCi/L	< 25.3	< 81.4	< 53.4	
Niobium-94	µCi/L	< 43.12	< 83.27	< 63.20	
RuRh-106	µCi/L	< 2981	< 6039	< 4510	
Cesium-134	µCi/L	< 44.33	< 92.4	< 68.4	
Cesium-137	µCi/L	312400	271700	292050	13.9%
Density	g/cm^3	1.39	1.42	1.41	2.1%

9 2 1 2 4 1 4 1 0 1 6

Constituent	Sample Number / Units	11a	11b	Mean	%Diff.
Aluminum	mg/L	9647	10967	10307	12.8%
Barium	mg/L	< 11.11	< 14.52	< 12.82	
Bismuth	mg/L	< 145.2	< 189.2	< 167.2	
Cadmium	mg/L	< 49.61	< 64.46	< 57.04	
Calcium	mg/L	94.38	99.44	96.91	5.2%
Chromium	mg/L	624.8	673.2	649.0	7.5%
Copper	mg/L	< 3.751	< 4.873	< 4.312	
Iron	mg/L	< 6.897	< 8.965	< 7.931	
Lead	mg/L	< 459.8	< 597.3	< 528.6	
Magnesium	mg/L	< 2.057	< 2.673	< 2.365	
Manganese	mg/L	< 55.66	< 72.38	< 64.02	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 66.55	< 86.46	< 76.51	
Phosphorus	mg/L	5511	6237	5874	12.4%
Potassium	mg/L	1023	1060.4	1042	3.6%
Silver	mg/L	< 3.63	< 4.719	< 4.17	
Sodium	mg/L	80080	96030	88055	18.1%
Titanium	mg/L	< 3.509	< 4.565	< 4.037	
Zinc	mg/L	< 9.438	< 12.32	< 10.88	
Zirconium	mg/L	< 25.63	< 36.19	< 30.91	
Carbonate	M	0.349		0.349	
Chloride	mg/L	2618	2783	2701	6.1%
Hydroxide	M	0.403		0.403	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	0.682	0.6666	0.674	2.3%
Nitrate	mg/L	77330	# 178200	76890	1.1%
Nitrate RR	mg/L		76450		

9 2 1 2 4 1 4 1 0 1 7

Constituent	Sample Number / Units	11a	11b	Mean	%Diff.
Phosphate	mg/L	17710	17380	17545	1.9%
Sulfate	mg/L	2464	2376	2420	3.6%
Cobalt-60	µCi/L	< 37.51	< 31.68	< 34.60	
Niobium-94	µCi/L	< 48.07	< 54.34	< 51.21	
RuRh-106	µCi/L	< 3498	< 3850	< 3674	
Cesium-134	µCi/L	< 49.83	< 61.71	< 55.77	
Cesium-137	µCi/L	214500	215600	215050	0.5%
Density	g/cm^3	1.39	1.42	1.41	2.1%

9 2 1 2 4 1 4 1 0 1 8

Constituent	Sample Number / Units	12a	12b	Mean	%Diff.
Aluminum	mg/L	21230	19470	20350	8.6%
Barium	mg/L	< 21.23	< 14.52	< 17.88	
Bismuth	mg/L	< 277.2	< 189.2	< 233.2	
Cadmium	mg/L	< 94.71	< 64.46	< 79.59	
Calcium	mg/L	166.1	158.4	162.3	4.7%
Chromium	mg/L	1177	1188	1183	0.9%
Copper	mg/L	< 7.161	5.621	6.391	
Iron	mg/L	< 13.2	< 8.965	< 11.1	
Lead	mg/L	< 877.8	< 597.3	< 737.6	
Magnesium	mg/L	< 3.927	< 2.673	< 3.300	
Manganese	mg/L	< 106.26	< 72.38	< 89.32	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 127.6	< 86.46	< 107.0	
Phosphorus	mg/L	1364	1474	1419	7.8%
Potassium	mg/L	2057	2123	2090	3.2%
Silver	mg/L	< 6.93	< 4.719	< 5.82	
Sodium	mg/L	154000	160600	157300	4.2%
Titanium	mg/L	< 6.699	< 4.565	< 5.632	
Zinc	mg/L	< 18.04	< 12.32	< 15.18	
Zirconium	mg/L	< 53.13	< 36.19	< 44.66	
Carbonate	M	0.72		0.72	
Chloride	mg/L	4829	# 49170	4851	0.9%
Chloride RR	mg/L		4873		
Hydroxide	M	1.16		1.16	
Fluoride	mg/L	< 11.0	< 11.0	< 11.0	
Nitrite	M	1.265	1.199	1.232	5.4%
Nitrate	mg/L	134200	138600	136400	3.2%

Constituent	Sample Number / Units	12a	12b	Mean	%Diff.
Phosphate	mg/L	3553	3564	3559	0.3%
Sulfate	mg/L	4433	4334	4384	2.3%
Cobalt-60	µCi/L	< 33.88	< 33.88	< 33.88	
Niobium-94	µCi/L	< 100.1	< 102.08	< 101.1	
RuRh-106	µCi/L	< 5918	< 5962	< 5940	
Cesium-134	µCi/L	< 99.77	< 99.77	< 99.77	
Cesium-137	µCi/L	427900	447700	437800	4.5%
Density	g/cm^3	1.35	1.35	1.35	0.0%

9 2 1 2 4 1 4 1 0 2 0

APPENDIX D

MEAN SAMPLE RESULT BY LOCATION

TANK 241-AN-106

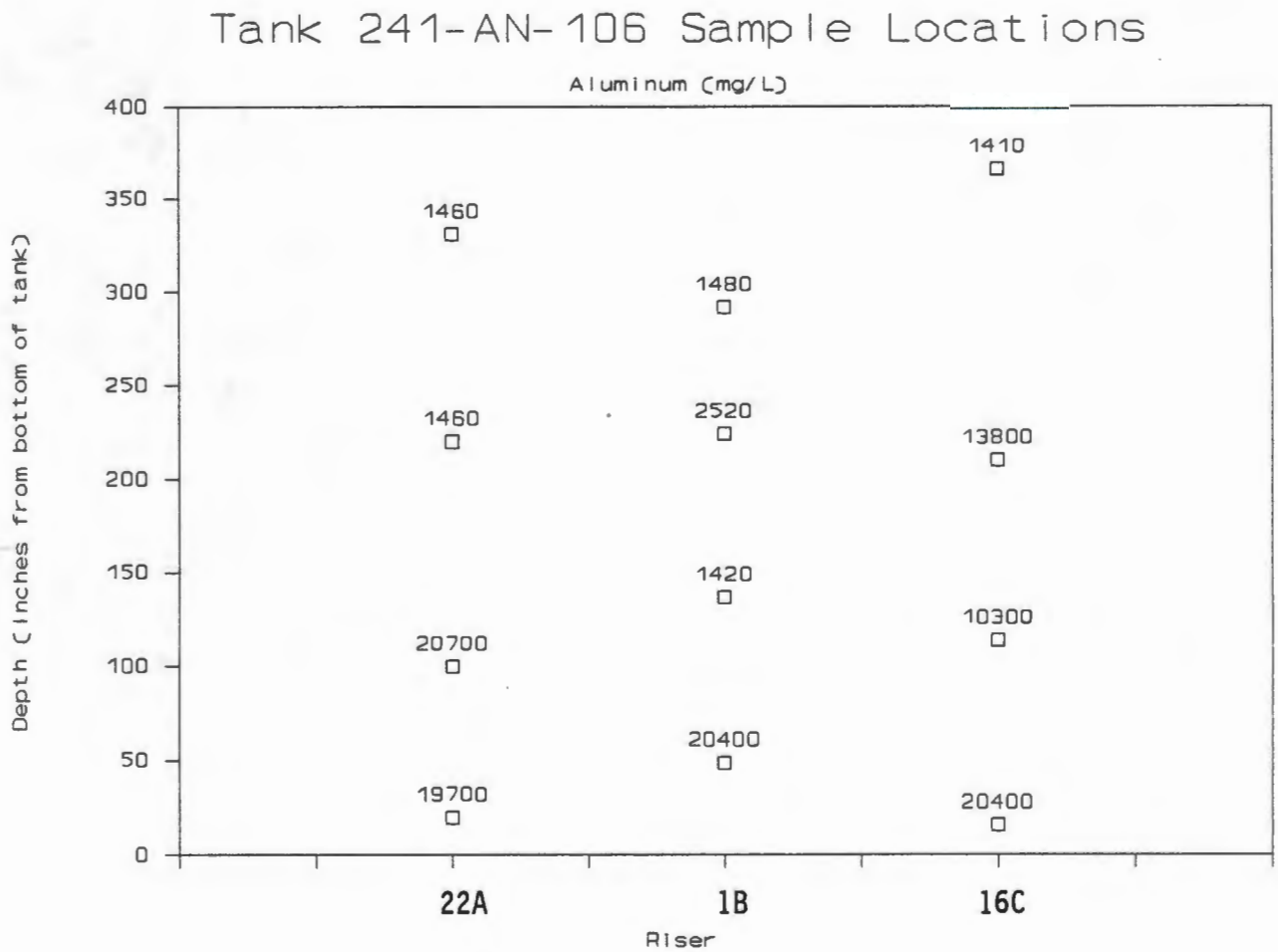
This page is intentionally left blank

Mean Value of Sample Tank 241-AN-106							
Constituent	Units	1	2	3	4	5	6
Aluminum	mg/L	1460	1460	20700	19700	1480	2520
Barium	mg/L	< 6.07	< 6.07	< 17.9	< 17.9	< 6.09	< 6.04
Bismuth	mg/L	< 79.2	< 79.2	< 233	< 233	< 79.3	< 79.3
Cadmium	mg/L	< 27.1	< 27.1	< 79.6	< 79.6	< 27.1	< 27.1
Calcium	mg/L	10.5	11.3	160	155	11.1	17.6
Chromium	mg/L	105	104	1150	1160	106	169
Copper	mg/L	< 2.05	< 2.05	< 6.02	6.18	< 2.03	< 2.03
Iron	mg/L	< 3.76	< 22.6	< 11.1	< 11.1	< 3.75	< 3.75
Lead	mg/L	< 251	< 251	< 738	< 738	< 251	< 251
Magnesium	mg/L	< 1.12	< 1.12	< 3.30	< 3.30	< 1.12	< 1.12
Manganese	mg/L	< 30.4	< 30.4	< 89.3	< 89.3	< 30.3	< 30.3
Mercury	mg/L	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Molybdenum	mg/L	< 36.3	< 51.4	< 107	< 107	< 36.3	< 36.3
Phosphorus	mg/L	10300	9720	1410	4700	9910	8060
Potassium	mg/L	294	323	2010	2010	292	401
Silver	mg/L	< 1.98	< 2.81	< 5.82	< 5.82	< 2.02	< 2.02
Sodium	mg/L	39500	40700	165000	164000	40200	42700
Titanium	mg/L	< 1.91	< 2.71	< 5.63	< 5.63	< 1.90	< 1.90
Zinc	mg/L	< 5.15	< 7.29	< 15.2	< 15.2	< 5.18	< 5.18

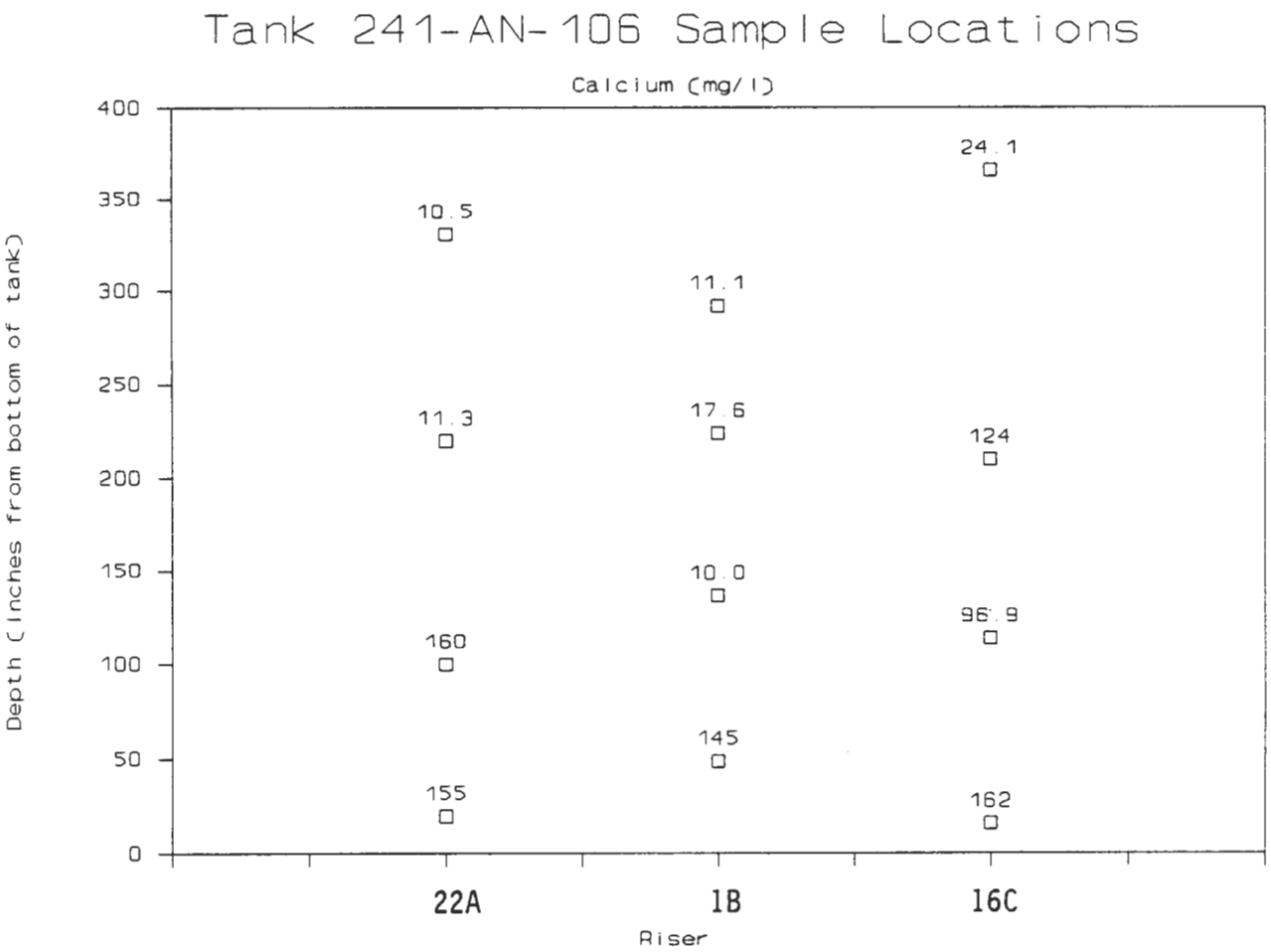
Mean Value of Sample Tank 241-AN-106							
Constituent	Units	1	2	3	4	5	6
Zirconium	mg/L	< 15.2	< 21.5	< 44.7	< 44.7	< 15.2	< 15.2
Carbonate	M	0.139	0.136	0.607	0.618	0.148	0.176
Chloride	mg/L	1160	1060	5430	4020	595	939
Hydroxide	M	0.087	0.091	1.11	0.782	0.089	0.142
Fluoride	mg/L	< 79.2	< 11.0	< 11.0	< 11.0	< 125	< 125
Nitrite	M	0.142	0.140	1.23	1.18	0.101	0.182
Nitrate	mg/L	17900	18300	162000	155000	16400	22300
Phosphate	mg/L	29600	29500	3450	4320	30100	25000
Sulfate	mg/L	2820	2710	4430	3650	499	840
Cobalt-60	μCi/L	9.16	< 12.7	< 46.1	< 38.3	< 2.28	12.3
Niobium-94	μCi/L	< 6.51	< 12.8	< 74.1	< 62.0	< 10.6	< 8.87
RuRh-106	μCi/L	< 383	< 898	< 5430	< 4240	< 444	< 521
Cesium-134	μCi/L	< 6.65	< 16.5	< 74.2	< 71.1	< 10.4	< 9.35
Cesium-137	μCi/L	42900	44100	439000	430000	42200	63400
Density	g/cm^3	1.11	1.11	1.37	1.60	1.12	1.13

Mean Value of Sample Tank 241-AN-106							
Constituent	Units	7	8	9	10	11	12
Aluminum	mg/L	1420	20400	1410	13800	10300	20400
Barium	mg/L	< 6.04	< 17.9	< 6.07	< 12.8	< 12.8	< 17.9
Bismuth	mg/L	< 79.3	< 233	< 79.2	< 167	< 167	< 233
Cadmium	mg/L	< 27.1	< 79.6	< 27.1	< 57.0	< 57.0	< 79.6
Calcium	mg/L	10.0	145	24.1	124	96.9	162
Chromium	mg/L	101	1170	106	831	649	1180
Copper	mg/L	< 2.03	< 6.02	< 2.05	< 4.31	< 4.31	6.39
Iron	mg/L	< 3.75	< 11.1	< 3.76	< 7.93	< 7.93	< 11.1
Lead	mg/L	< 251	< 738	< 251	< 529	< 529	< 738
Magnesium	mg/L	< 1.12	< 3.30	2.71	< 2.37	< 2.37	< 3.30
Manganese	mg/L	< 30.3	< 89.3	< 30.4	< 64.0	< 64.0	< 89.3
Mercury	mg/L	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Molybdenum	mg/L	< 36.3	< 107	< 36.3	< 76.5	< 76.5	< 107
Phosphorus	mg/L	9150	1360	9600	3680	5870	1420
Potassium	mg/L	308	1940	288	1250	1040	2090
Silver	mg/L	< 2.02	< 5.82	< 1.98	< 4.17	< 4.17	< 5.82
Sodium	mg/L	37200	155000	38400	104000	88100	157000
Titanium	mg/L	< 1.90	< 5.63	< 1.91	< 4.04	< 4.04	< 5.63
Zinc	mg/L	< 5.18	< 15.2	< 5.15	< 10.9	< 10.9	< 15.2

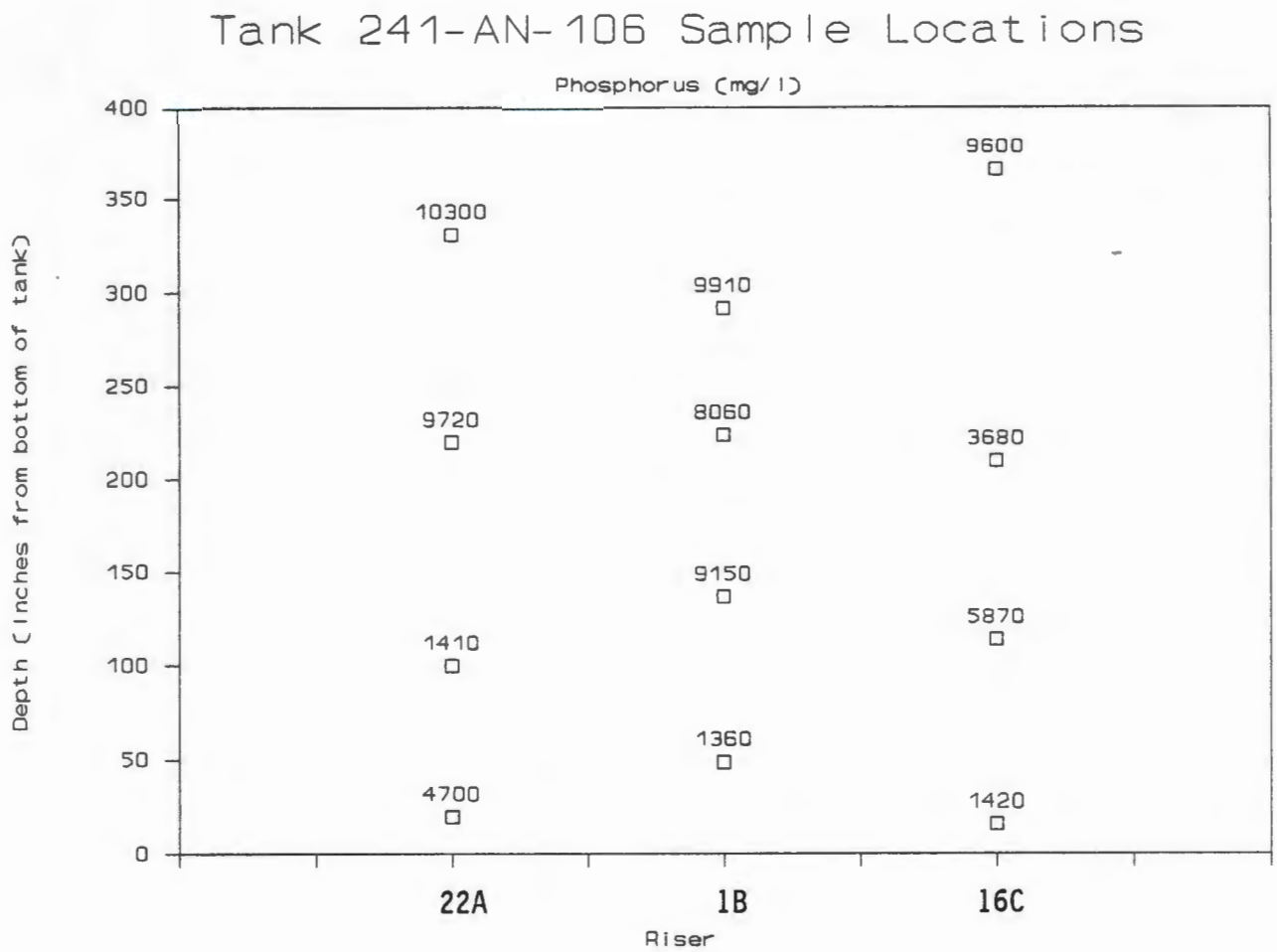
Mean Value of Sample Tank 241-AN-106							
Constituent	Units	7	8	9	10	11	12
Zirconium	mg/L	< 15.2	< 44.7	< 14.7	< 32.0	< 30.9	< 44.7
Carbonate	M	0.134	0.551	0.178	0.443	0.349	0.720
Chloride	mg/L	685	4700	646	3290	2700	4850
Hydroxide	M	0.072	1.12	0.245	0.508	0.403	1.16
Fluoride	mg/L	< 111	< 11.0	< 11.0	< 11.0	< 11.0	< 11.0
Nitrite	M	0.097	1.26	0.141	0.837	0.674	1.23
Nitrate	mg/L	15000	166000	16200	95800	76900	136000
Phosphate	mg/L	27500	3790	29300	10800	17500	3560
Sulfate	mg/L	534	4570	893	3120	2420	4380
Cobalt-60	µCi/L	< 5.63	< 37.4	7.76	< 53.4	< 34.6	< 33.9
Niobium-94	µCi/L	< 10.4	< 60.3	< 6.40	< 63.2	< 51.2	< 101
RuRh-106	µCi/L	< 704	< 4120	< 431	< 4510	< 3670	< 5940
Cesium-134	µCi/L	< 11.5	< 63.6	< 6.71	< 68.4	< 55.8	< 99.8
Cesium-137	µCi/L	41400	446000	42900	292000	215000	438000
Density	g/cm^3	1.09	1.36	1.12	1.41	1.41	1.35



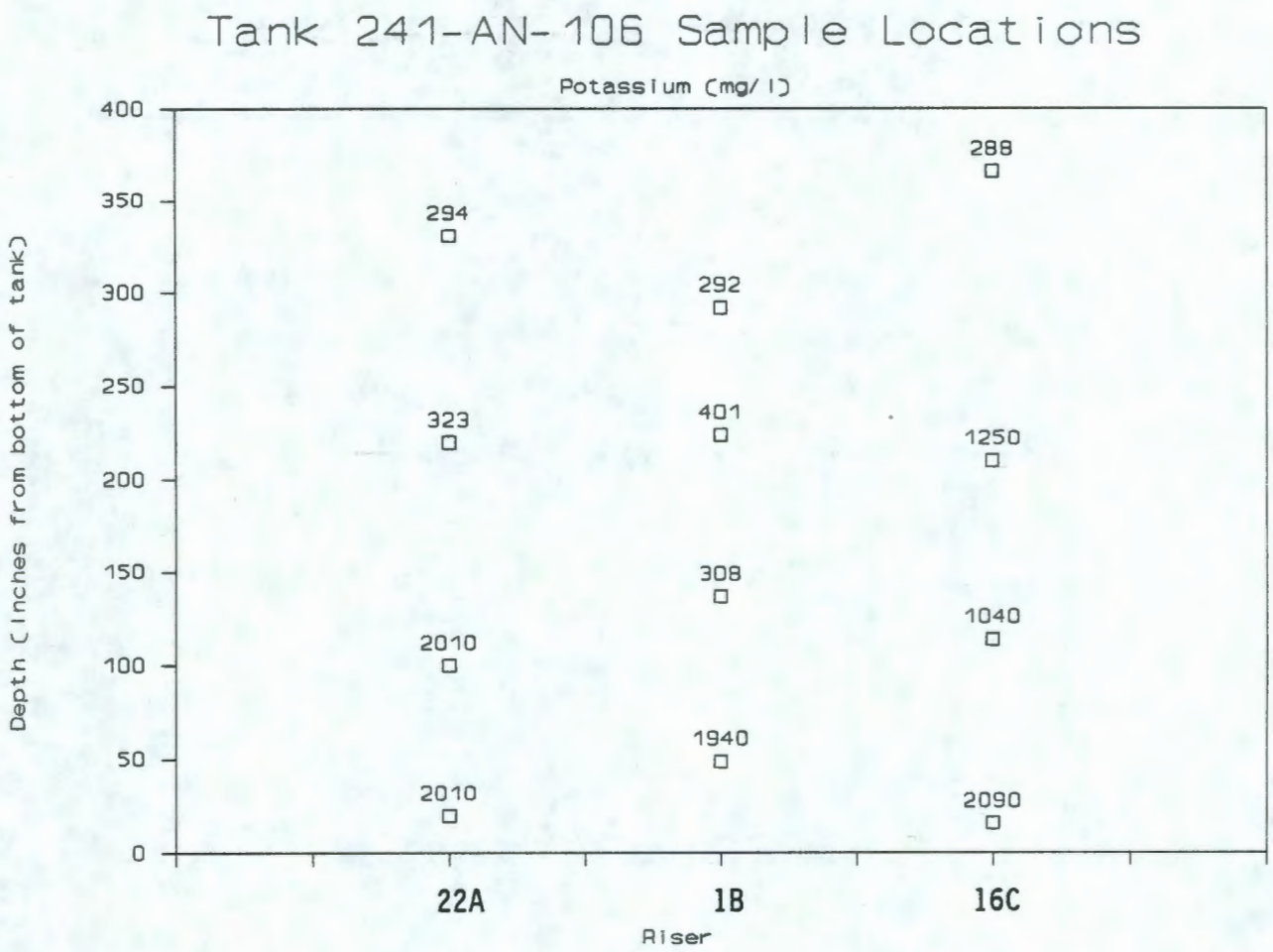
Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°



Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

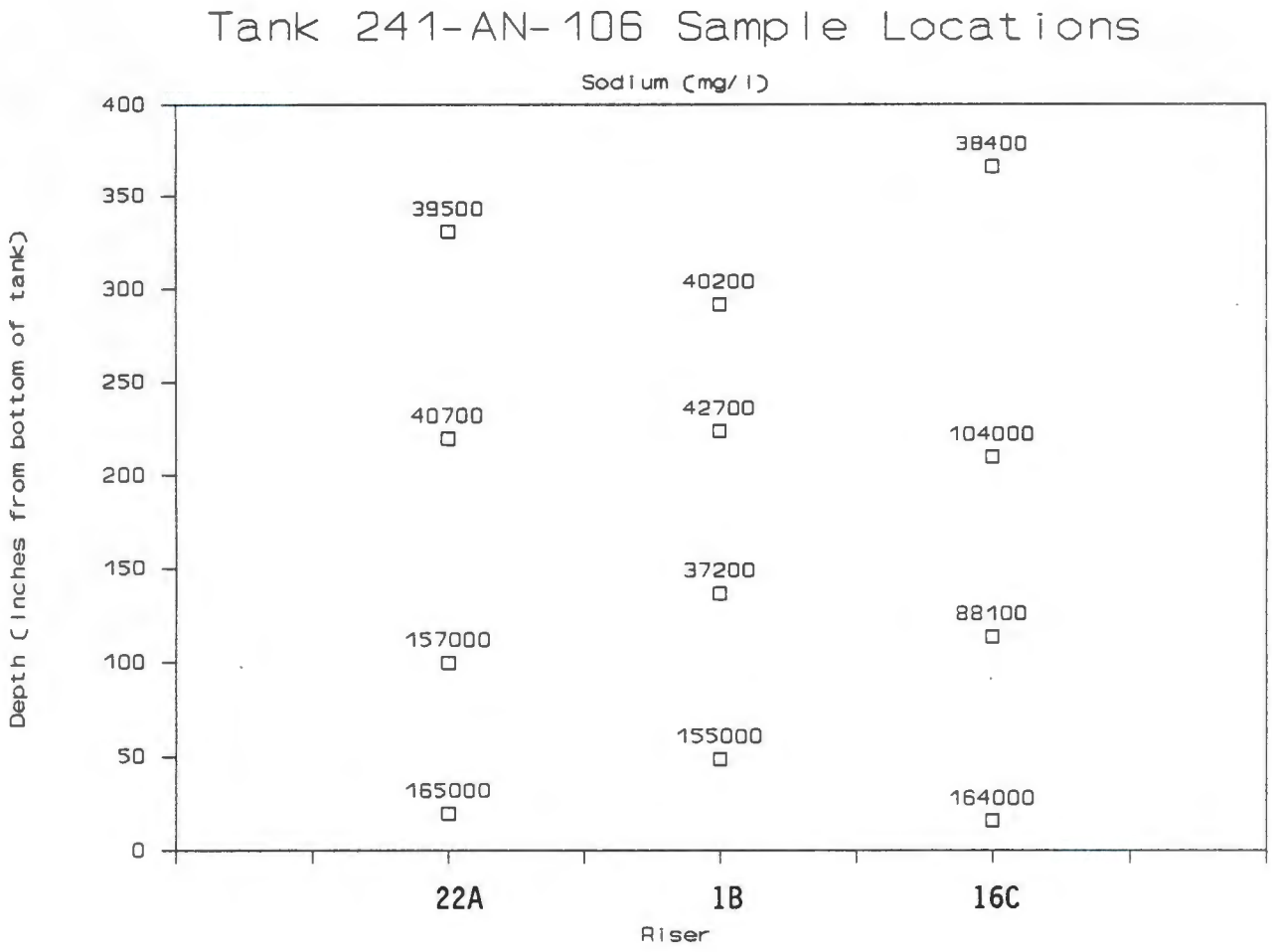


Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

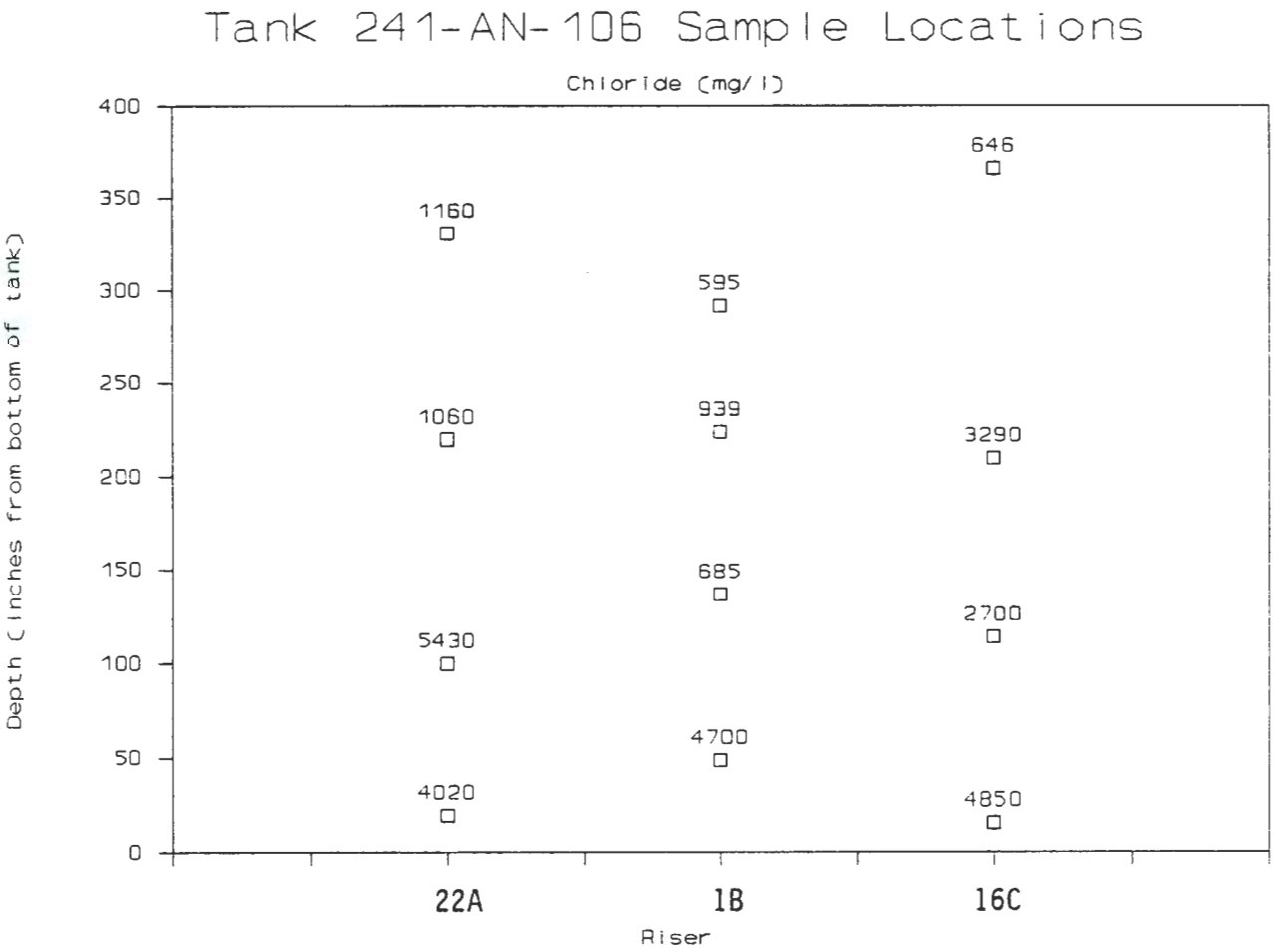


Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

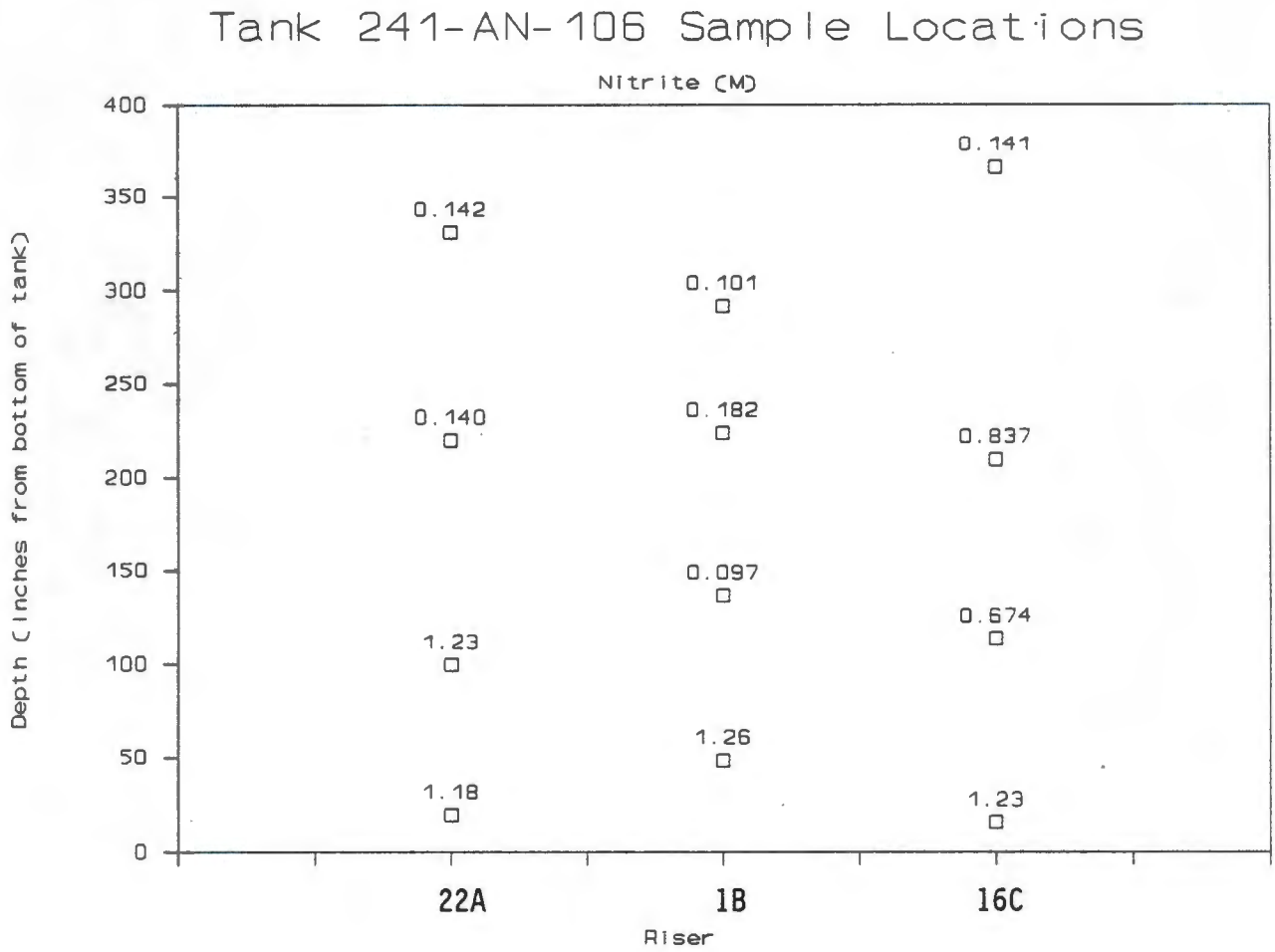
9 2 1 2 4 1 4 1 0 3 1



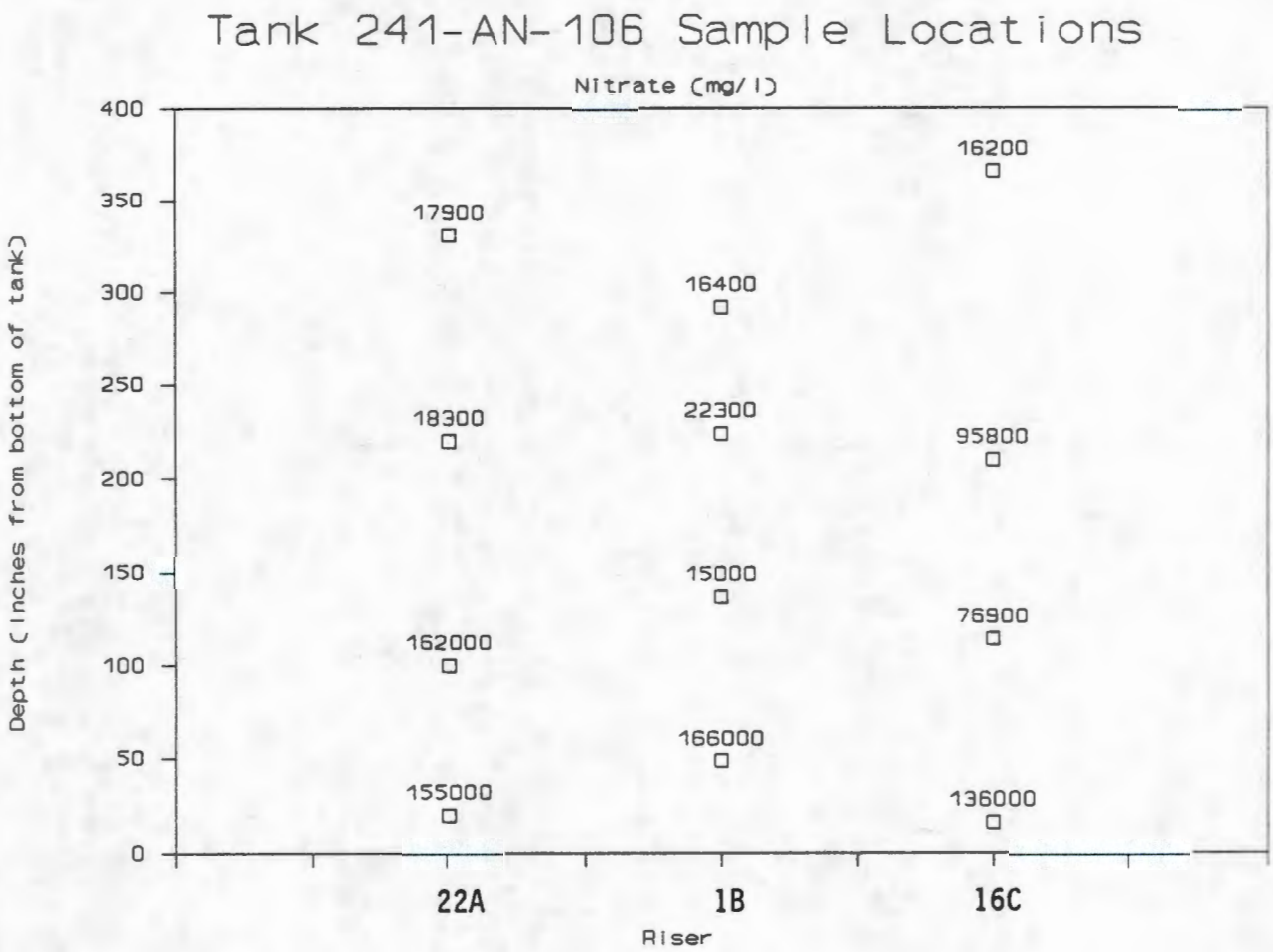
Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°



Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

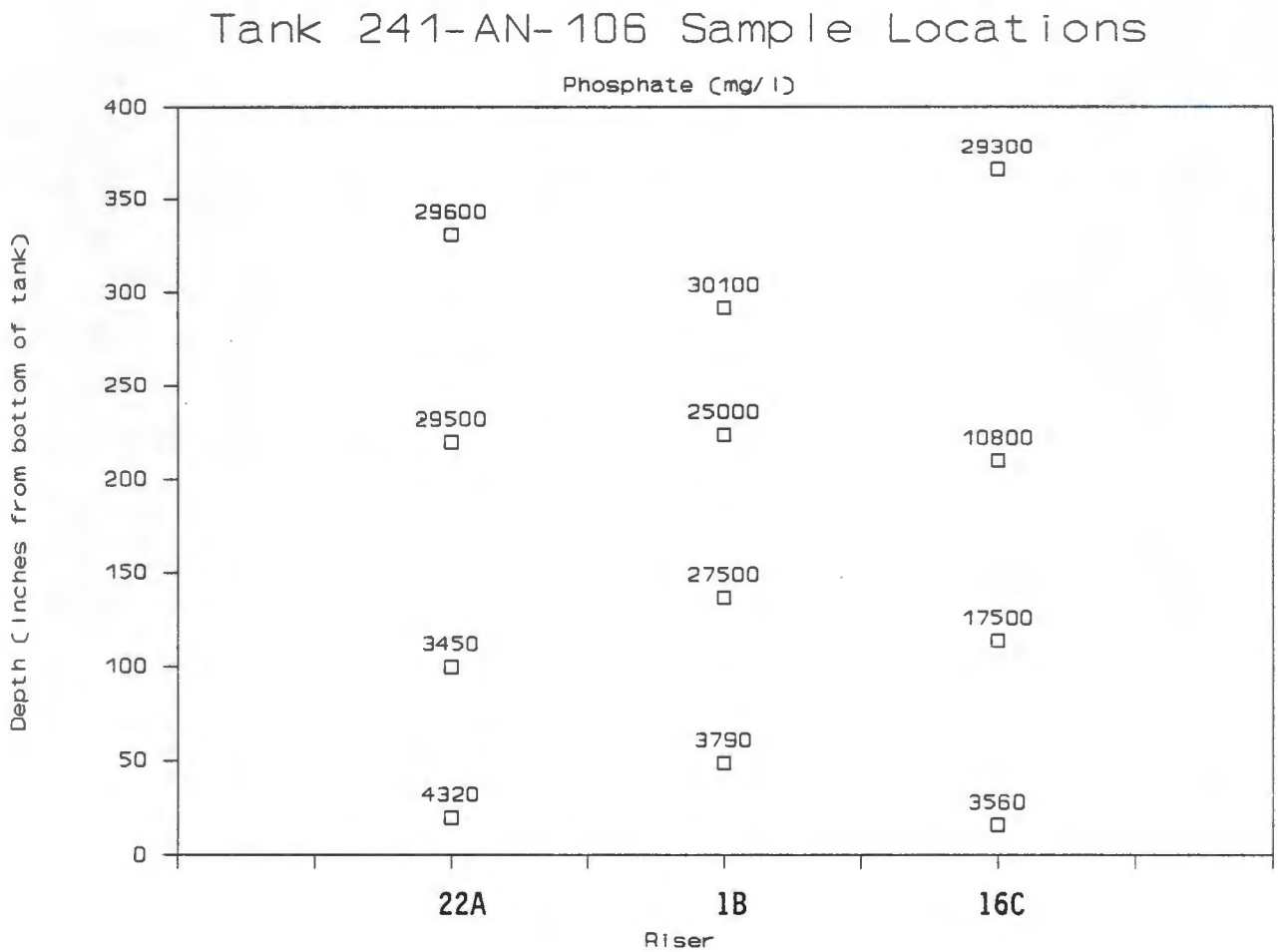


Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

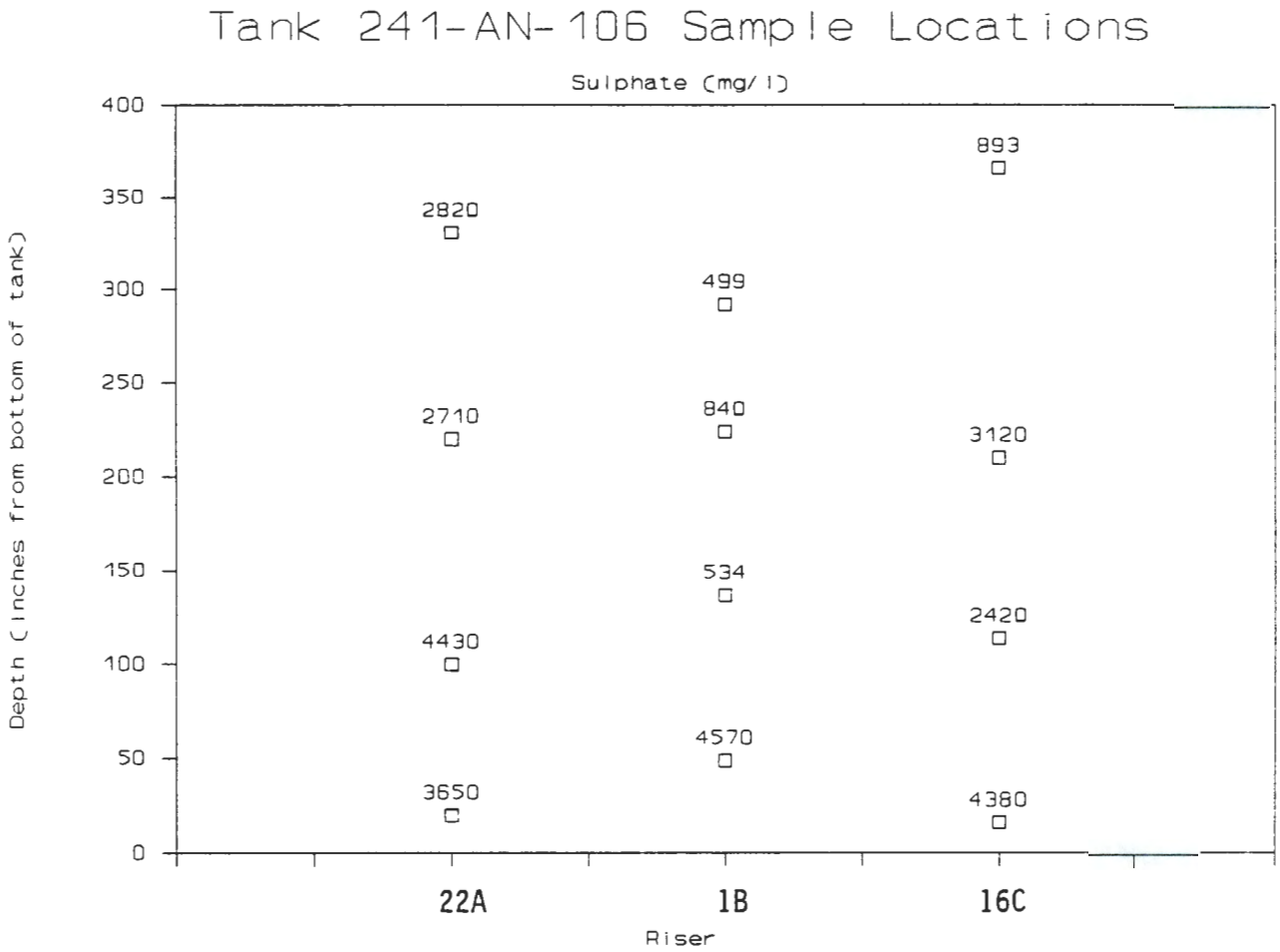


Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

9 2 1 2 4 1 4 1 0 3 5

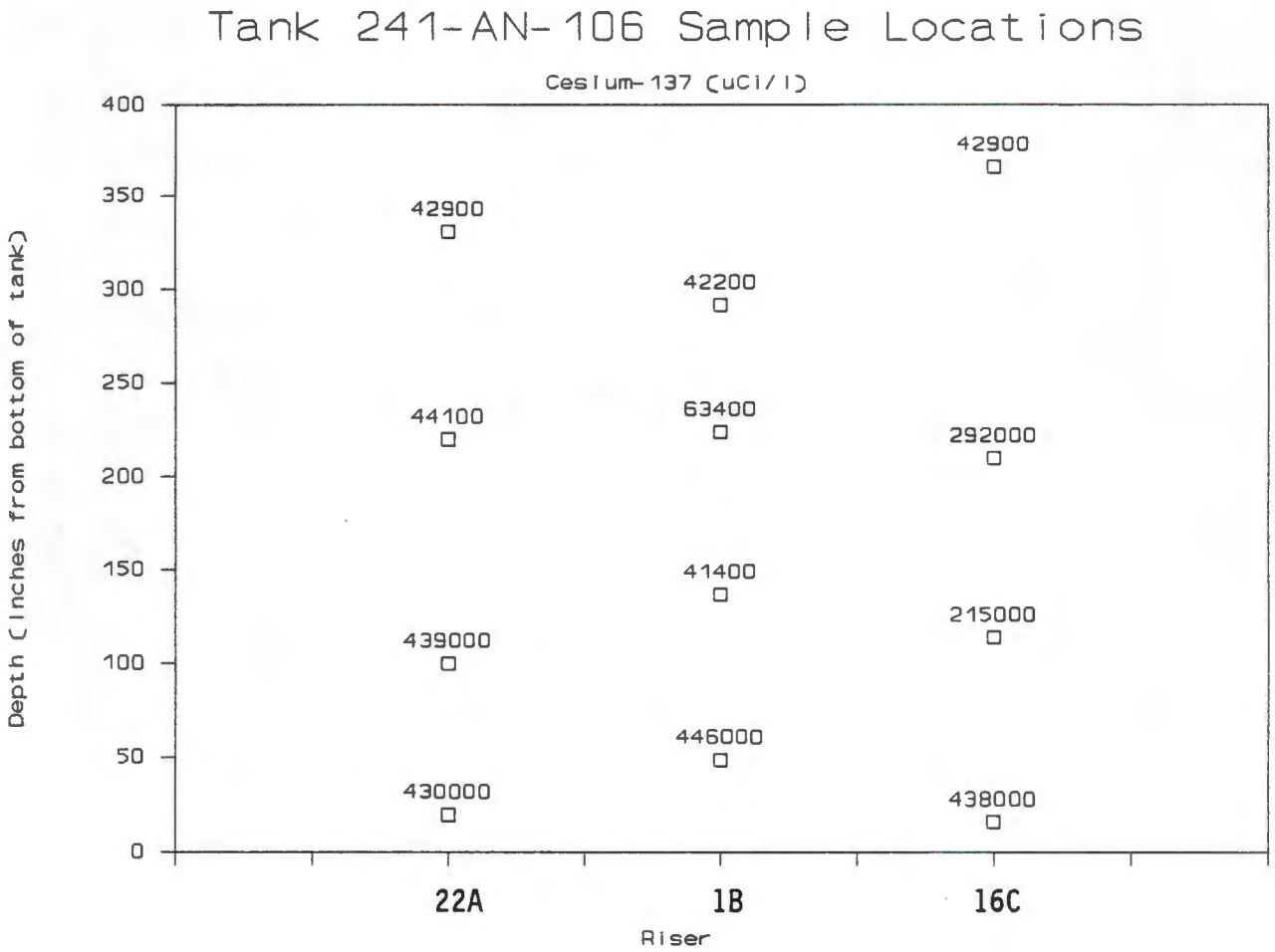


Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

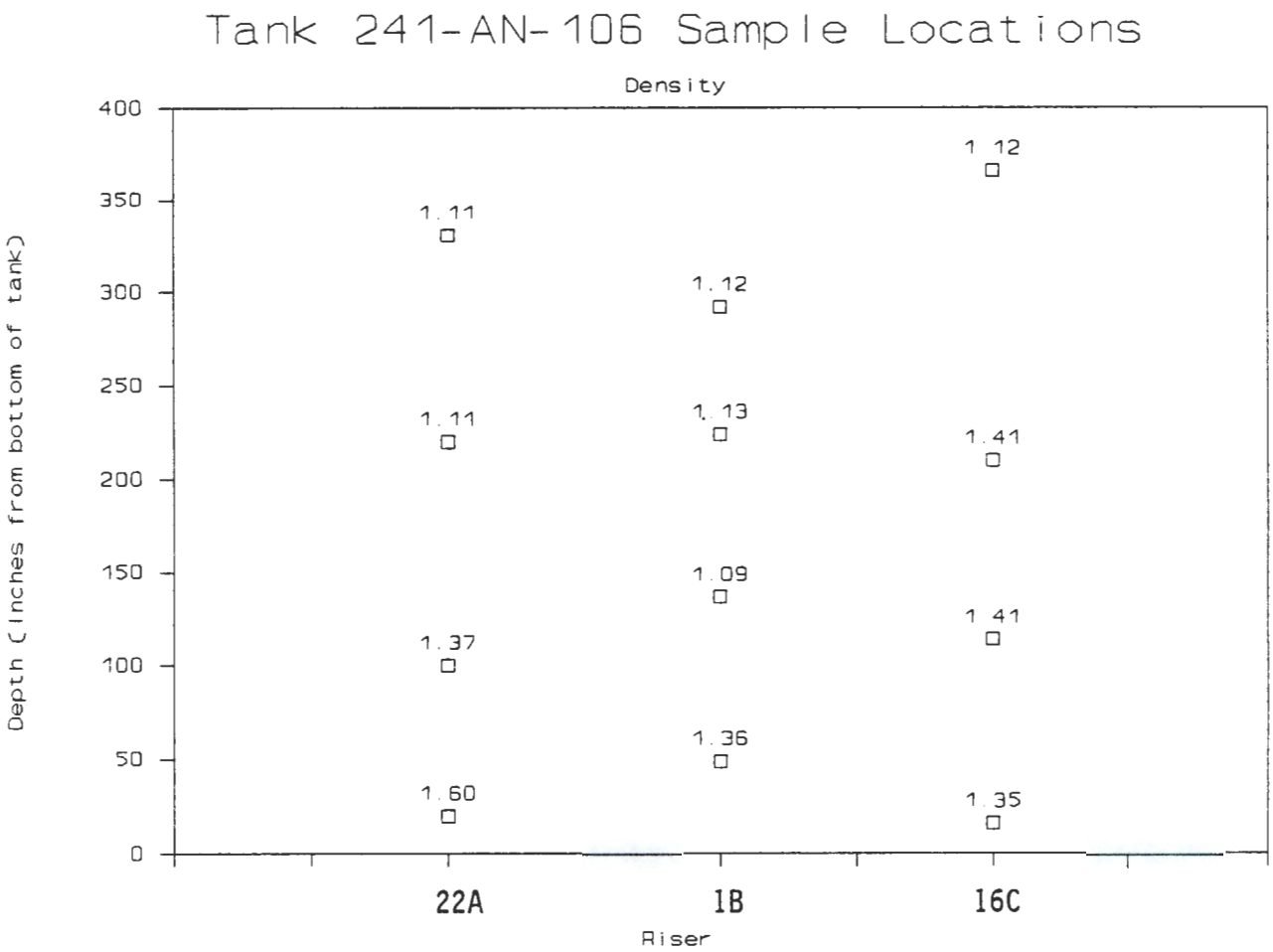


Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

9 2 1 2 4 1 1 0 3 7



Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°



Riser Number	Radius	Angle
22A	10 feet	180°
1B	20 feet	280°
16C	28 feet	65°

9 2 1 2 4 1 4 1 0 3 9

APPENDIX E

FPLSD RESULTS

TANK 241-AN-106

This page is intentionally left blank

Analysis of Variance indicates whether or not the treatment means; e.g. in our case location is the treatment, are significantly different. Multiple comparison procedures are then used to determine which means are significantly different. Fisher's protected least significant difference (FPLSD) technique is as follows:

> If the absolute value of the difference between any two treatment means exceeds the LSD, the effects of the two treatment means are judged to be significantly different; if the absolute value of the difference does not exceed the LSD, no such conclusion is reached.

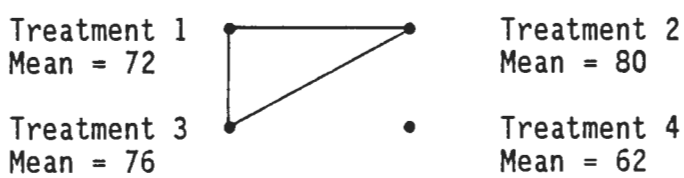
This technique is used only if the "F" statistic from the ANOVA indicates significant differences between means; i.e. "protected".

FPLSD results are usually presented in tabular form. Suppose the following table represents an FPLSD for some experimental data.

Treatment	Mean	FPLSD
4	62	A
1	72	B
3	76	B
2	80	B

The results can be interpreted as follows. If the treatment means have a letter in common, then there is no significant difference between the means. For the given example, the means for Groups 1, 2, and 3 are not significantly different, but the mean for Group 4 is significantly different from the other three means.

FPLSD results are also expressed in graphic form. If the treatment means are not significantly different, then a line is drawn connecting the two means. For the example presented above, the following represents the FPLSD results.



Constituent	Sample Number	Mean Value	FPLSD	Constituent	Sample	Mean	FPLSD
Aluminum	9	1410	A	Potassium	9	288	A
	7	1420	A		5	292	A
	1	1460	A		1	294	A
	2	1460	A		7	308	A
	5	1480	A		2	323	A
	6	2520	A		6	401	A
	11	10300	B		11	1040	B
	10	13800	C		10	1250	C
	4	19700	D		8	1940	D
	8	20400	D		3	2010	DE
	12	20400	D		4	2010	DE
	3	20700	D		12	2090	E

Constituent	Sample Number	Mean Value	FPLSD	Constituent	Sample	Mean	FPLSD
Calcium	7	10.0	A	Sodium	7	37200	A
	1	10.5	A		9	38400	A
	5	11.1	A		1	39500	A
	2	11.3	A		5	40200	A
	6	17.6	A		2	40700	A
	9	24.1	A		6	42700	A
	11	96.9	B		11	88100	B
	10	124	C		10	104000	B
	8	145	D		8	155000	C
	4	155	D		12	164000	C
	3	160	D		4	165000	C
	12	162	D		3	157000	C

Constituent	Sample Number	Mean Value	FPLSD	Constituent	Sample	Mean	FPLSD
Chromium	7	101	A	Chloride	5	595	A
	2	104	A		9	646	A
	1	105	A		7	685	AB
	5	106	A		6	939	BC
	9	106	AB		2	1060	C
	6	169	B		1	1160	C
	11	649	C		11	2700	D
	10	831	D		10	3290	E
	3	1150	E		4	4020	F
	4	1160	E		8	4700	G
	8	1170	E		12	4850	G
	12	1180	E		3	5430	H

Constituent	Sample Number	Mean Value	FPLSD	Constituent	Sample	Mean	FPLSD
Phosphorus	8	1360	A	Nitrite	7	0.097	A
	3	1410	A		5	0.101	AB
	12	1420	A		2	0.140	BC
	10	3680	B		9	0.141	C
	4	4700	C		1	0.142	CD
	11	5870	D		6	0.182	D
	6	8060	E		11	0.674	E
	7	9150	F		10	0.837	F
	9	9600	FG		4	1.18	G
	2	9720	FG		3	1.23	H
	5	9910	FG		12	1.23	H
	1	10300	G		8	1.26	H

Constituent	Sample Number	Mean Value	FPLSD	Constituent	Sample	Mean	FPLSD
Nitrate	7	15000	A	Cesium-137	7	41400	A
	9	16200	A		5	42200	AB
	5	16400	A		1	42900	AB
	1	17900	A		9	42900	AB
	2	18300	A		2	44100	AB
	6	22300	A		6	63400	B
	11	76900	B		11	215000	C
	10	95800	C		10	292000	D
	12	136000	D		4	430000	E
	4	155000	E		12	438000	EF
	3	162000	EF		3	439000	EF
	8	166000	F		8	446000	F

Constituent	Sample Number	Mean Value	FPLSD	Constituent	Sample	Mean	FPLSD
Phosphate	3	3450	A	Density	7	1.09	A
	12	3560	A		1	1.11	A
	8	3790	A		2	1.11	A
	4	4320	A		5	1.12	A
	10	10800	B		9	1.12	A
	11	17500	C		6	1.13	A
	6	25000	D		12	1.35	B
	7	27500	E		8	1.36	BC
	9	29300	F		3	1.37	BC
	2	29500	F		10	1.41	C
	1	29600	F		11	1.41	C
	5	30100	F		4	1.60	D

Constituent	Sample Number	Mean Value	FPLSD
Sulphate	5	499	A
	7	534	A
	6	840	B
	9	893	B
	11	2420	C
	2	2710	D
	1	2820	D
	10	3120	E
	4	3650	F
	12	4380	G
	3	4430	GH
	8	4570	H

9 2 1 2 4 1 4 1 0 4 9

APPENDIX F

CALCULATIONS TO DETERMINE THE COMPOSITE TANK 241-AN-106

This page is intentionally left blank

From: Process Chemistry & Engineering Laboratories 12711-89-128
Phone: 3-2475/3-5285 2704S/200W T6-18
Date: November 7, 1989
Subject: STATISTICAL ANALYSIS OF THE ANALYTICAL RESULTS FOR THE INDIVIDUAL SAMPLES FROM TANK 241-AN-106

To: R. D. Claghorn R1-48

cc: S. O. DeLeon S1-52
D. A. Dodd T6-50
A. P. Hammitt T6-50
L. Jensen T6-18
J. P. Sloughter T6-07
J. A. Voogd R1-48
CMS/TLW-File/LB

Reference: SD-WM-TP-061, Rev. 1, 241-AN-106 Characterization and Grout Formulation Hot Cell Test Plan, Released June 22, 1989.

Samples were taken (April 1989) from Tank 241-AN-106 to test analytical procedures used in the hot cell and to characterize the contents of the tank (see reference for details). Samples from 12 locations (as determined by the Statistics Team according to a stratified random sampling plan) were transferred to Analytical Systems Laboratory (ASL). Subsamples were obtained and submitted to 222-S Analytical Laboratories Facilities for analysis. Concentrations of 33 analytes were determined for each subsample. The results were then provided to the Statistics Team for evaluation.

According to the test plan, a single composite will be made from the 12 original samples. The composite must be representative of the tank's contents after the waste has been mixed. The purpose of the statistical analysis of the individual sample data was to determine the significance of the concentration differences between the 12 samples. This information would then be used to determine the portion of each sample required to develop the composite sample.

The results from only three analytes (Cesium-137, Phosphate, Aluminum) are presented here, one from each of the three multi-element analytical methods (Gamma Energy Analysis, Dionex, Inductively Coupled Plasma Spectroscopy). These analytes were chosen because they are considered important variables in the grout process.

Hanford Operations and Engineering Contractor for the US Department of Energy

The duplicate analyses for each analyte measured were compared with the limits based on standards performance data (see reference for details). This comparison was done to ensure the validity of the analytical results. Since standards data is expected to show less variability than the actual sample data, the duplicates were also compared to limits based on actual sample data. If the duplicate analysis exceeded both limits and the analyte was considered important to the grout process, then the sample pair was resubmitted for confirmatory analysis.

For almost two-thirds of the analytes measured (Lead, Bismuth, Zirconium, Iron, Barium, Cadmium, Copper, Magnesium, Manganese, Molybdenum, Silver, Zinc, Titanium, Fluoride, Cobalt-60, Cesium-134, Ruthenium-Rhodium-106, Mercury, and Niobium-94) every analytical value reported was a "less than" value. Standard statistical analyses could not be performed with these non-numerical results. Since these analytes were considered to be of less importance to the grout process, the samples were not resubmitted for analysis. However, it is recommended that analytical laboratory personnel use the individual sample data results ("less than" values) to determine the appropriate dilution factors for each analyte requested for the analysis of the composite samples. The objective of this action is to obtain fewer "less than" values for the composite samples.

A two-way analysis of variance (ANOVA) was performed to detect differences within a section (horizontal layer of the tank) and differences between sections. The results from the ANOVA indicated significant differences within a section and between sections for all analytes reported with actual values. The significant differences between sections indicate that the tank contents are stratified while the significant differences within a section indicate that the interface between the sections is not well defined.

A one-way ANOVA, using each sample location as a class, was performed to provide more detail about the tank contents. The results from the ANOVA for each analyte indicated significant differences between the 12 sample locations. A multiple comparison procedure known as Fisher's Protected Least Significant Difference (FPLSD) was used to compare means based on the analytical results and to determine which sample locations would then be significantly different. In general, the FPLSD results showed two layers with no distinct division (see Figures 1, 2, and 3). The data and FPLSD results for Cesium-137, Phosphate, and Aluminum are presented in Tables 1 and 2, respectively.

The analytical data indicated that Tank 241-AN-106 appears to have two layers and that the division is somewhere between 100 and 200 inches from the bottom (see Figures 1, 2, and 3). In order to determine the composite formulation, the overall tank concentration of an analyte was calculated assuming two layers and several different strata divisions. The majority of these divisions were at depths between 100 and 200 inches from the bottom of the tank, however several other depths were incorporated for completeness. This included a strata depth of 376 inches from the bottom of the tank to represent the "no stratification" option.

The calculations were performed using two methodologies. The first approach adhered to the original stratified random sampling plan when considering the two layers. It can be mathematically shown that no matter where the division between the two layers occurs, the same overall tank concentration and variability will be reached. The second approach assumed total randomization within the two layers. This approach requires an assumption because even though the samples were drawn with total randomization within a section, there is not total randomization within the two layers determined by the strata divisions. However, if there is reasonable belief that the same sample locations would have been chosen under total randomization within the two layers, then the assumption is valid.

The variance associated with each overall tank concentration was computed for each method of calculation. For those calculations using the second approach, 95% Confidence Intervals were estimated using the Satterthwaite's approximation to determine the degrees of freedom for the t-statistic. The overall tank concentrations, the standard deviations, and the 95% Confidence Intervals (if appropriate) for Cesium-137, Phosphate, and Aluminum are listed in Table 3 and illustrated in Figures 4, 5, and 6.

Student-Newman-Keuls (SNK) multiple comparison test was used to compare the overall tank concentration based on stratification to the overall tank concentrations based on total randomization within the two layers. To perform the SNK procedure an estimate of the experimental error variance is required. The smallest variance from the overall tank concentration calculations was chosen to estimate this variance in order to keep the test conservative. The results of the SNK test for Cesium-137, Phosphate, and Aluminum indicated that no significant differences exist between any of the estimates of the overall tank concentration.

Since the overall tank concentration estimates were not significantly different, then it is reasonable to believe that the 241-AN-106 tank contents can be represented by compositing an equal volume of each of the 12 samples received from the tank. This composite can then be used to proceed with the formulation verification testing required by the test plan.

The results of this letter were provided verbally to R. D. Claghorn and A. P. Hammitt prior to preparation of the composite samples.

If you have any questions, please call.

C M Sager

C. M. Sager
Scientist

T L Welsh

T. L. Welsh
Senior Statistician

mcr

Attachments

Table 1

Sample Number	Riser	Depth[1]	Cs-137 (uCi/l)	PO4 (mg/l)	Al (mg/l)
R5232	1B	292"	41910	30800	1496
R5235			42570	29480	1460
R5233	1B	224"	63580	25960	2574
R5238			63250	24090	2453
R5234	1B	137"	41910	27940	1392
R5239			42570	27060	1445
R5274	1B	49"	444400	3773	20020
R5278			446600	3806	20680
R5275	16C	366"	42240	29040	1364
R5279			43560	29480	1463
R5276	16C	210"	312400	10637	12430
R5282			271700	10923	15070
R5277	16C	114"	214500	17710	9647
R5285			215600	17380	10967
R5290	16C	16"	427900	3553	21230
R5296			447700	3564	19470
R5291	22A	331"	42350	29480	1452
R5297			43450	29810	1463
R5292	22A	220"	46310	29370	1485
R5298			41910	29590	1441
R5293	22A	100"	434500	1067	19910
R5299			444400	1010	21450
R5294	22A	20"	415800	4180	18700
R5300			431200	4466	20790

[1] inches from bottom of tank

9 2 1 2 4 1 4 1 0 5 5

Table 2

Riser	Depth[2]	Location	Cs-137 (uCi/l)	PO4 (mg/l)	Al (mg/l)
16C	366"	5	AB	A	A
22A	331"	9	AB	A	A
1B	292"	1	AB	A	A
1B	224"	2	AB	A	A
22A	220"	10	D	E	A
16C	210"	6	B	C	C
1B	137"	3	EF	G	A
16C	114"	7	A	B	B
22A	100"	11	C	D	D
1B	49"	4	E	F	D
22A	20"	12	EF	F	D
16C	16"	8	F	F	D

The results from a multiple comparison test can be interpreted as follows. If locations have a letter in common, then there is no significant difference between the analytical results. For example, for Aluminum, the analytical results for locations 5, 9, 1, 2, 10, 3 are not significantly different. In Figures 1, 2, 3 the locations which cannot be statistically distinguished from each other are represented by connecting line segments.

[2] depth in inches from bottom of tank

Table 3

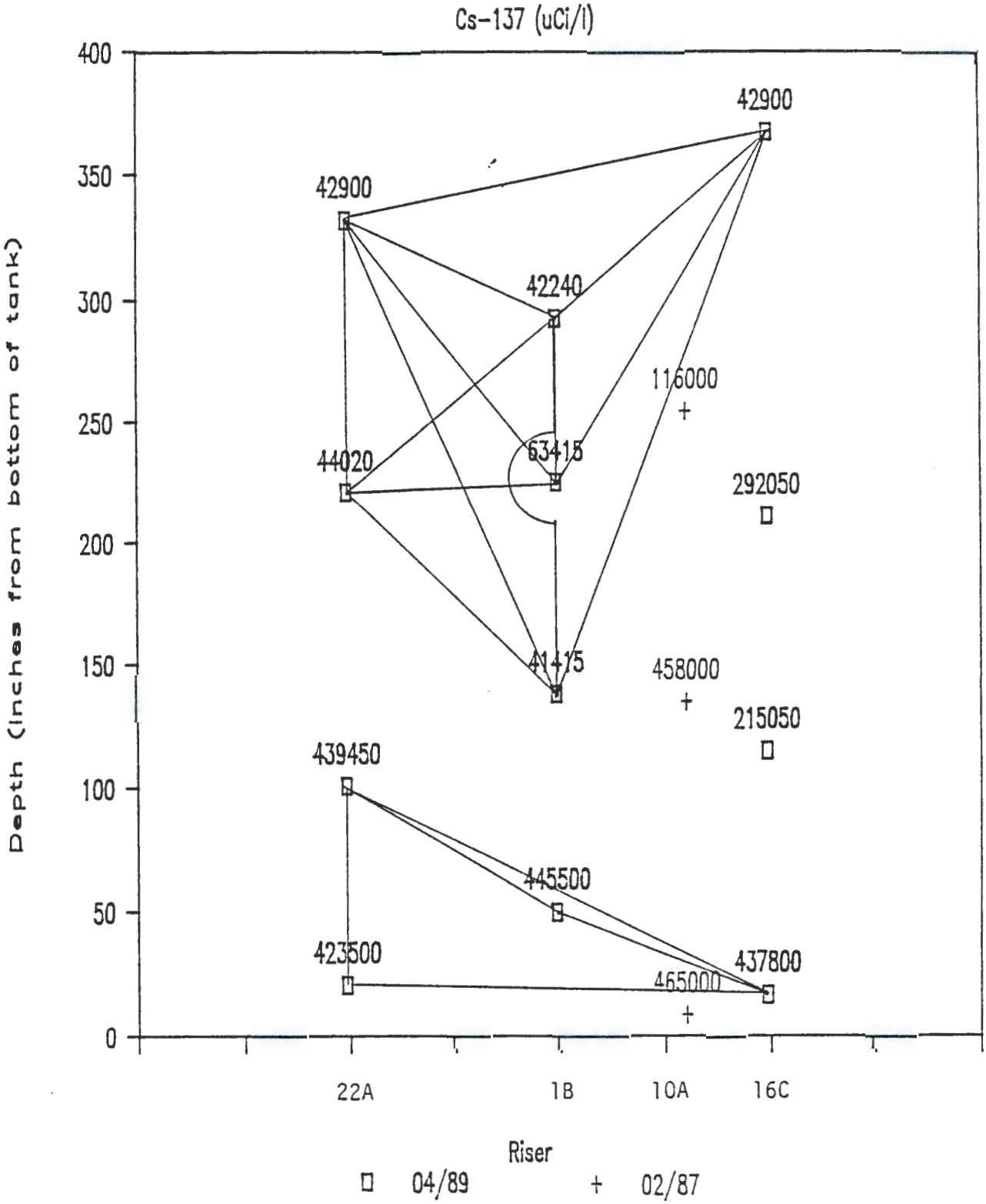
Analyte	Sampling Technique[3]	Strata Depth[4]	Tank Concentration	Standard Deviation[5]	95% Confidence Interval Lower Limit	Upper Limit
Cs-137	SRS	25	0.1844	0.0500	0.0714	0.2974
	SRS	50	0.1758	0.0423	0.0782	0.2734
	SRS	100	0.1880	0.0256	0.1290	0.2470
	SRS	125	0.1846	0.0278	0.1227	0.2465
	SRS	150	0.1860	0.0369	0.1038	0.2681
	SRS	175	0.2023	0.0388	0.1146	0.2901
	SRS	200	0.2187	0.0413	0.1234	0.3139
	SRS	218	0.2098	0.0339	0.1268	0.2929
	SRS	376	0.2109	0.0532	0.0937	0.3280
	STRAT		0.2109	0.0351		
PO4	SRS	25	19.325	3.358	11.729	26.921
	SRS	50	19.825	2.992	12.926	26.724
	SRS	100	19.140	1.846	14.774	23.506
	SRS	125	19.354	2.001	14.827	23.880
	SRS	150	19.302	2.526	13.675	24.929
	SRS	175	18.232	2.593	12.455	24.009
	SRS	200	17.162	2.705	10.925	23.399
	SRS	218	17.744	2.145	12.494	22.993
	SRS	376	17.674	3.501	9.969	25.378
	STRAT		17.674	2.391		
Al	SRS	25	8.318	2.398	2.894	13.743
	SRS	50	7.927	2.069	3.156	12.699
	SRS	100	8.494	1.277	5.473	11.515
	SRS	125	8.318	1.338	5.292	11.345
	SRS	150	8.384	1.777	4.424	12.343
	SRS	175	9.168	1.859	4.962	13.374
	SRS	200	9.952	1.971	5.406	14.497
	SRS	218	9.527	1.602	5.608	13.446
	SRS	376	9.577	2.548	3.968	15.186
	STRAT		9.577	1.704		

[3] SRS = Simple Random Sampling STRAT = Stratified Random Sampling

[4] inches from bottom of tank

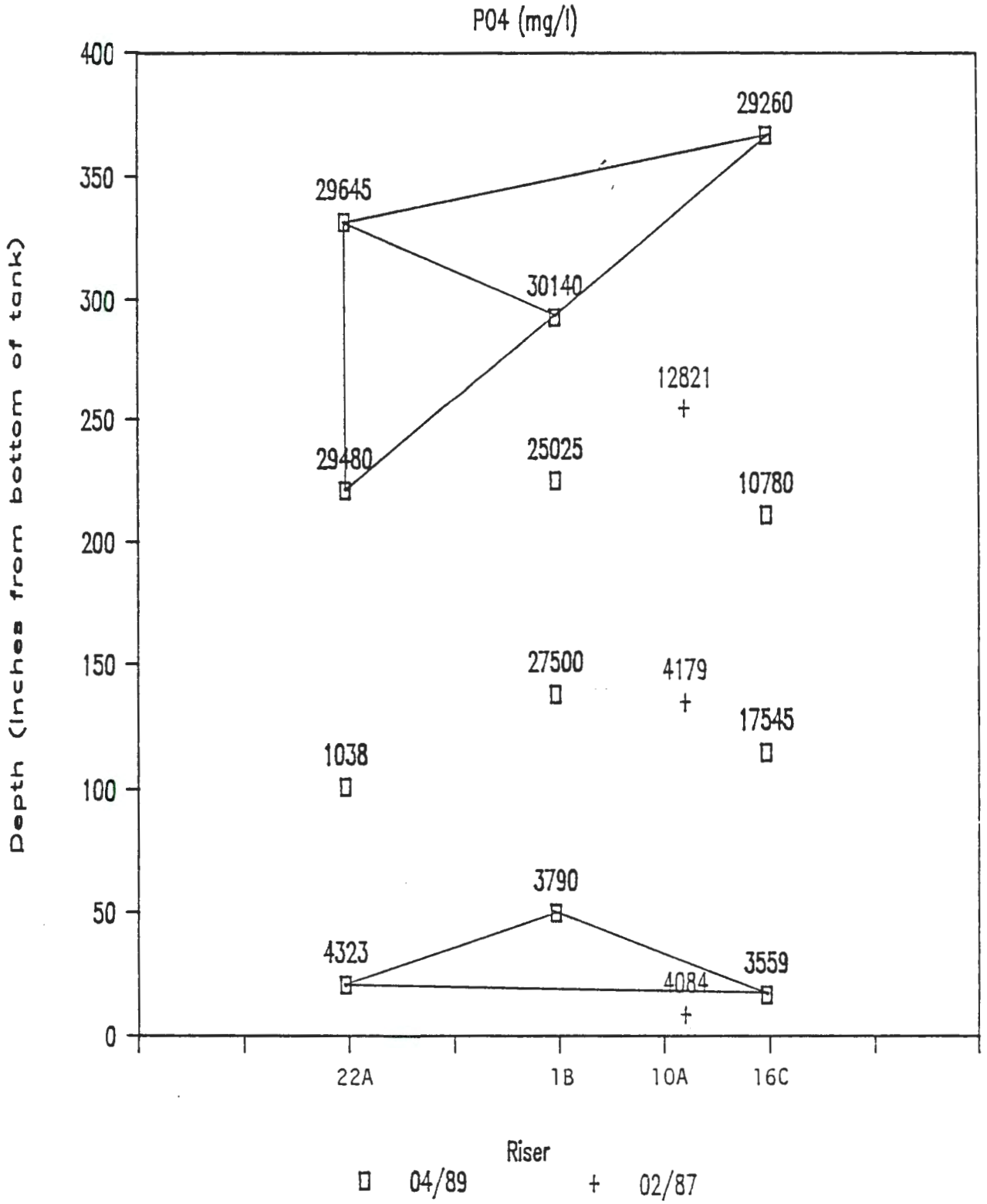
[5] the square root of the variance calculation

TK-241-AN-106 Characterization Data



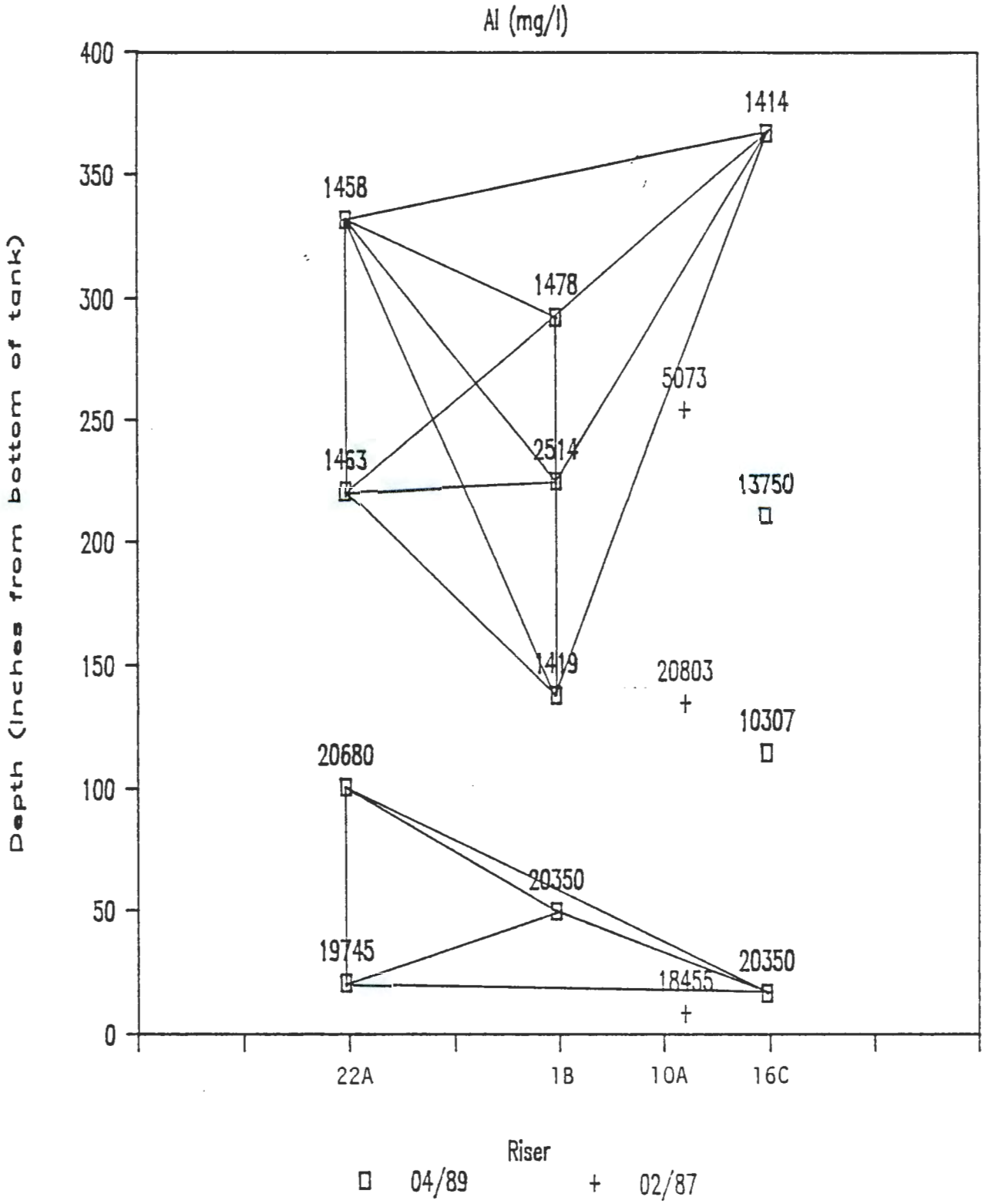
- The value of each data point is the average of two (replicate) measurements.
- Historical data (02/87) are included in the graph for reference only, the statistical analyses were performed on the 04/89 data.
- The risers are ordered with respect to distance from the center of the tank. Riser 22A is 10' from the center while 1B is 20' from the center. Risers 16C and 10A are 28' from the center but on opposite sides of the tank.

TK-241-AN-106 Characterization Data

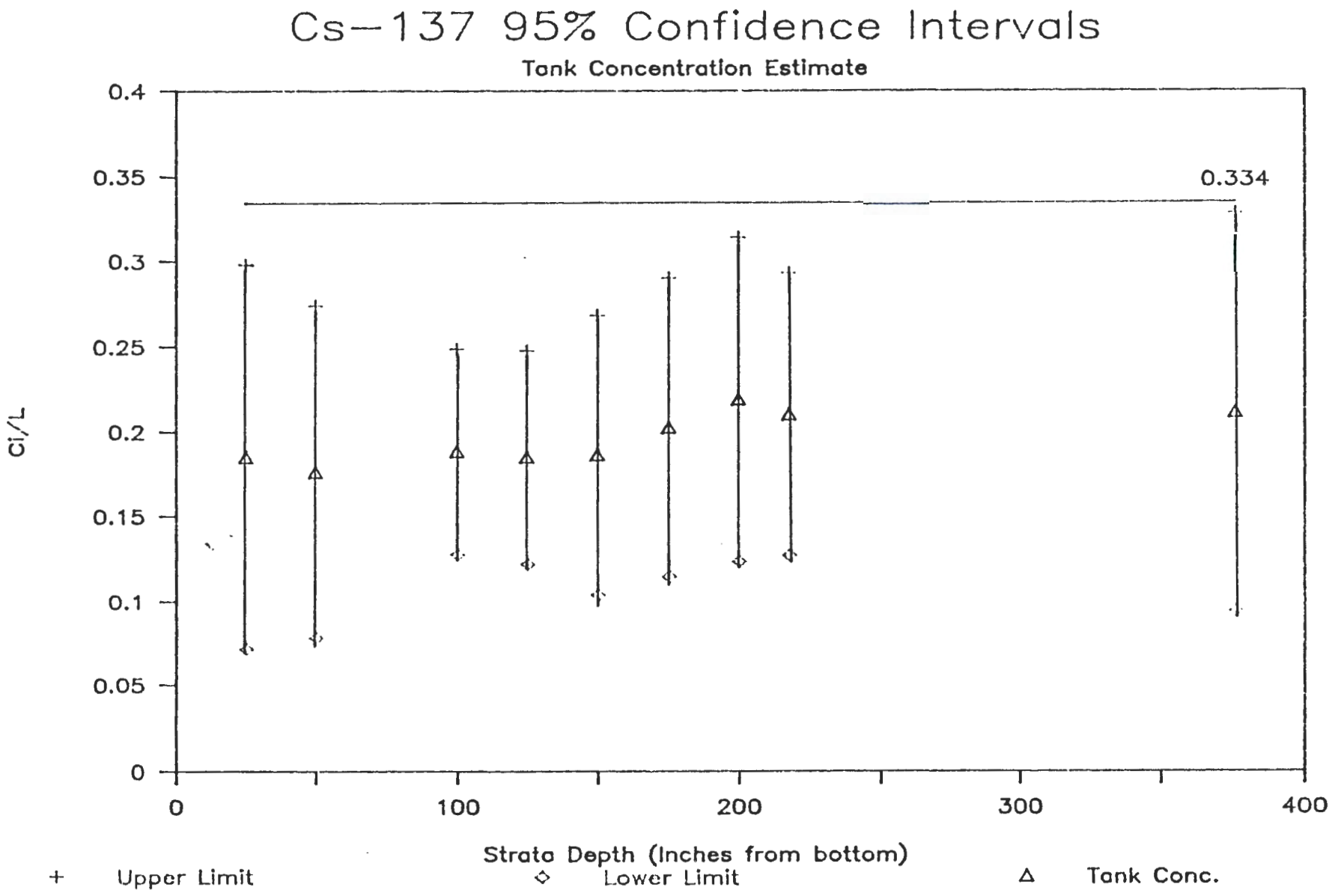


- The value at each data point is the average of two (replicate) measurements.
- Historical data (02/87) are included in the graph for reference only, the statistical analyses were performed on the 04/89 data.
- The risers are ordered with respect to distance from the center of the tank. Riser 22A is 10' from the center while 1B is 20' from the center. Risers 16C and 10A are 28' from the center but on opposite sides of the tank.

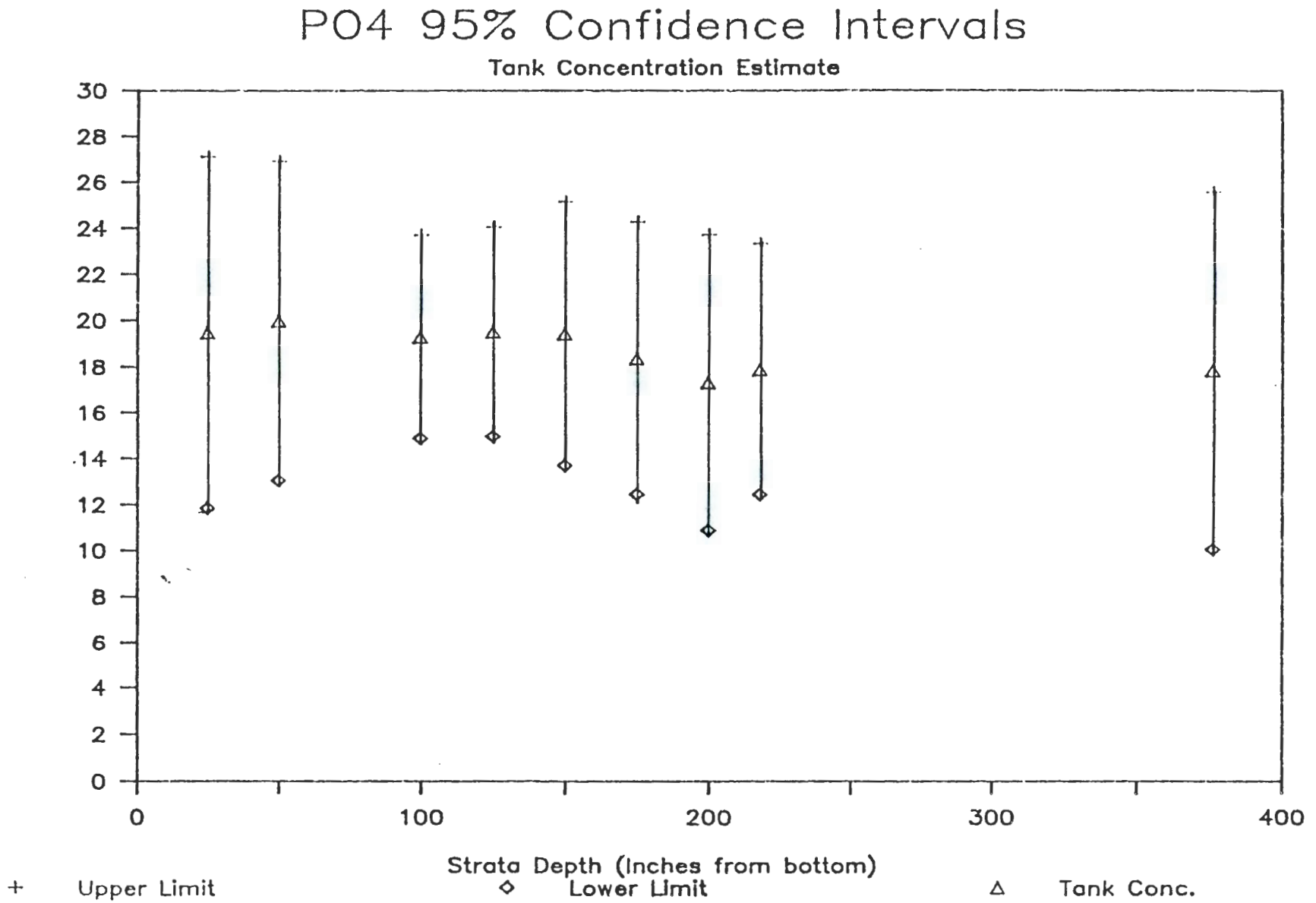
TK-241-AN-106 Characterization Data



- The value of each data point is the average of two (replicate) measurements.
- Historical data (02/87) are included in the graph for reference only, the statistical analyses were performed on the 04/89 data.
- The risers are ordered with respect to distance from the center of the tank. Riser 22A is 10' from the center while 1B is 20' from the center. Risers 16C and 10A are 28' from the center but on opposite sides of the tank.



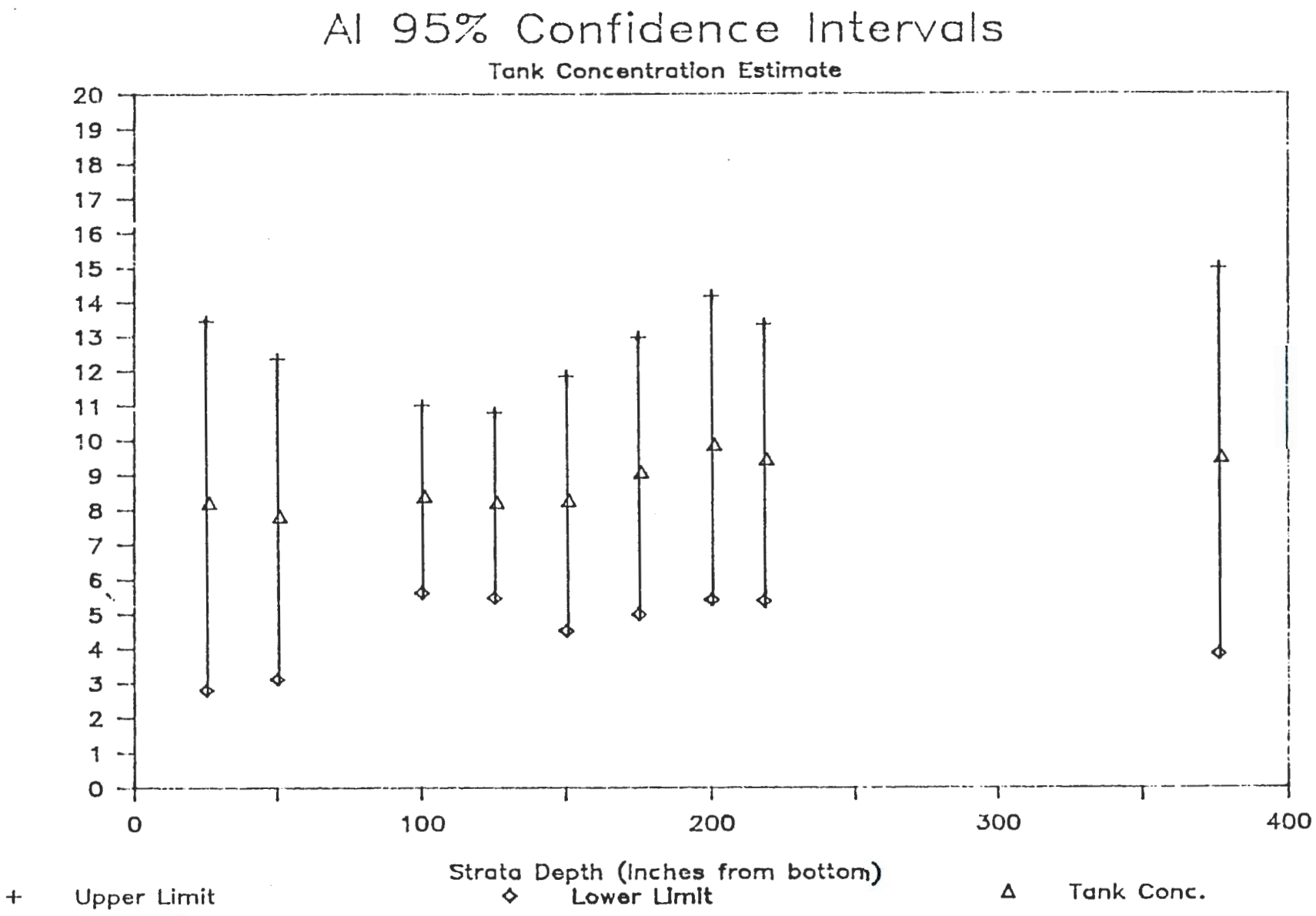
- The y-axis represents the estimate of the overall tank concentration based on the presence of a strata division at the depth represented on the x-axis.
- A 95% Confidence Interval for each estimated overall tank concentration was computed. The upper and lower limits are illustrated.
- The overall tank concentration estimate at 376.1 inches from the bottom of the tank was based on equal volumes.



- The y-axis represents the estimate of the overall tank concentration based on the presence of a strata division at the depth represented on the x-axis.
- A 95% Confidence Interval for each estimated overall tank concentration was computed. The upper and lower limits are illustrated.
- The overall tank concentration estimate at 376.1 inches from the bottom of the tank was based on equal volumes.

Figure 6

12711-89-128



- The y-axis represents the estimate of the overall tank concentration based on the presence of a strata division at the depth represented on the x-axis.
- A 95% Confidence Interval for each estimated overall tank concentration was computed. The upper and lower limits are illustrated.
- The overall tank concentration estimate at 376.1 inches from the bottom of the tank was based on equal volumes.

This page is intentionally left blank

9 2 1 2 4 1 4 1 0 6 4

9 2 1 2 4 1 4 1 0 6 5

APPENDIX G

COMPOSITE RESULTS

TANK 241-AN-106

9 2 1 2 4 1 4 1 0 6 6

This page is intentionally left blank

Constituent	Sample Number / Units	Comp 1	Comp 2	Mean	%Diff.
Aluminum	mg/L	9042	9526	9284	5.2%
Arsenic	mg/L	0.1002	0.0446	0.0724	76.8%
Barium	mg/L	< 11.11	< 11.11	< 11.11	
Bismuth	mg/L	< 145.2	< 145.2	< 145.2	
Cadmium	mg/L	< 49.61	< 49.61	< 49.61	
Calcium	mg/L	86.24	94.16	90.20	8.8%
Chromium	mg/L	557.7	569.8	563.8	2.1%
Copper	mg/L	< 3.751	< 3.751	< 3.751	
Iron	mg/L	< 6.897	< 6.897	< 6.897	
Lead	mg/L	< 459.8	< 459.8	< 459.8	
Magnesium	mg/L	3.179	2.376	2.778	28.9%
Manganese	mg/L	< 55.66	< 55.66	< 55.66	
Mercury	mg/L	< 0.05	< 0.05	< 0.05	
Molybdenum	mg/L	< 66.55	< 66.55	< 66.55	
Phosphorus	mg/L	6006	6215	6111	3.4%
Potassium	mg/L	1067	1093.4	1080	2.4%
Selenium	mg/L	0.182	0.0862	0.134	71.4%
Silver	mg/L	< 3.63	< 3.63	< 3.63	
Sodium	mg/L	89980	90640	90310	0.7%
Titanium	mg/L	< 3.509	< 3.509	< 3.509	
Uranium	g/L	0.004	0.00412	0.004	3.1%
Zinc	mg/L	< 9.438	< 9.438	< 9.438	
Zirconium	mg/L	< 27.83	< 27.83	< 27.83	
Ammonia	mg/L	154	89.1	123	51.8%
Ammonia RR	mg/L	156	93.3		
Carbonate	M	0.334	0.32	0.33	4.3%
Chloride	mg/L	2299	2629	2464	13.4%

9 2 1 2 4 1 1 0 5 7

Constituent	Sample Number / Units	Comp 1	Comp 2	Mean	%Diff.
Cyanide	g/L	0.00548	0.0057	0.0056	3.9%
Hydroxide	M	0.45	0.498	0.47	10.1%
Hydroxide RR	M	0.456	0.506		
Fluoride	mg/L	< 111.1	< 12.1	< 61.6	
Nitrite	M	0.6385	0.6504	0.6445	1.8%
Nitrate	mg/L	69190	67760	68475	2.1%
Phosphate	mg/L	17820	18920	18370	6.0%
Sulfate	mg/L	2079	2222	2151	6.6%
Tritium	µCi/L	4.06	2.82	3.44	36.0%
Carbon-14	µCi/L	0.252	0.418	0.335	49.6%
Cobalt-60	µCi/L	< 6.204	< 12.1	< 9.2	
Selenium-79	µCi/L	0.236	0.244	0.240	3.3%
Strontium-90	µCi/L	2260	2200	2230	2.7%
Niobium-94	µCi/L	< 19.25	< 26.84	< 23.05	
Tc-99	µCi/L	77.2	61.2	69.2	23.1%
RuRh-106	µCi/L	< 1188	< 1595	< 1392	
Iodine-129	µCi/L	< 0.0788	< 0.072	< 0.075	
Cesium-134	µCi/L	< 21.45	< 25.96	< 23.71	
Cesium-137	µCi/L	203500	205700	204600	1.1%
Np-237	g/L	< 0.00306	< 0.00306	< 0.00306	
Pu-238	µCi/L	0.0206	0.01882	0.0197	9.0%
Pu-239/240	µCi/L	0.0428	0.0328	0.0378	26.5%
Am-241	µCi/L	0.616	0.596	0.606	3.3%
Curium-243	µCi/L	0.0464	0.047	0.047	1.3%
TOC	mg/L	3340	3180	3260	4.9%
Water	wt%	78.3	76.96	77.6	1.7%
Density	g/cm^3	1.22	1.23	1.23	0.8%

9 2 1 2 4 1 1 0 6 8

9 2 1 2 4 1 4 1 0 6 9

APPENDIX H

ORGANIC RESULTS

TANK 241-AN-106

This page is intentionally left blank

ORGANIC COMPOUND ANALYSIS REPORT
Volatiles

SAMPLE ANALYSIS REPORTED

Analysis of liquid waste samples for volatile organic compounds by methods specified in the EPA Contract Laboratory Program Statement of Work (CLP 2/88) are the subject of this report.

SAMPLE DESCRIPTION

The sample identified by the client as "241-AN 106 Composite" and labeled as "APH-361" was received at the 325 building Analytical Hot Cells on 8/29/90. No chain of custody papers accompanied the sample. The sample was contained in a 125 ml clear glass bottle with black, paper lined caps used for the sample closures. The sample bottle was approximately 60% full and 40% headspace, and was received at room temperature. The sample containment and storage observations were not consistent with those specified in "USEPA Contract Laboratory Program, Statement of Work for Organic Analysis", February 1988.

The sample was entered in the sample log and assigned the Chemical Measurements Laboratory number "90-6940". The sample was split on 8/31/90 by transferring approximately 10 ml into a pre-cleaned 20 ml volatile sample bottle with a septa closure and transferred to storage at 4° celsius. The remaining sample was stored at room temperature in the analytical hot cell to be extracted for semi-volatile components later. Refrigerated storage of highly radioactive waste samples is extremely limited so the aliquot of sample for volatile analysis is given priority.

Due to the sample containment and storage prior to laboratory receipt, factors such as holding time, temperature, and headspace for the volatile aliquot were no longer considered to be as critical.

SAMPLE PREPARATION

The sample was screened for volatile compounds by performing hexadecane extractions followed by analysis on a gas chromatograph/ flame ionization detector (GC/FID). Sample and solvent quantities were modified from the CLP screening procedure. Four mls of sample and 2 ml of hexadecane were used. Standards were made by addition of 20 ul of desired compounds in methanol to blank water. Screen standard levels were 2000 ug/l for each compound.

Screen data indicated that the sample did not require dilution for analysis on the gas chromatograph/ mass spectrometer (GC/MS) instrument for volatile compounds.

In anticipation of foaming problems during the purging procedures, a second purge vessel was installed in line after the purge vessel containing the sample.

Samples were prepared for GC/MS analysis according to the EPA-CLP method. To reduce foaming problems, one ml of sample was diluted to 5 mls by using blank water. Surrogates and spikes were used during the purging procedure according to the protocol.

ANALYSIS METHOD

Hexadecane extracts for volatile screens were analyzed directly following extraction as described above. The screening analysis employed a HP-5890 GC (WB60701) located in lab 330, 325 building.

GC/MS analysis was performed using a HP-5890/5970 GC/MS (WB38464) and is described as system 2 in lab 427, 325 building.

Both the GC/FID volatile screens and GC/MS volatile analysis were performed per the Environmental Protection Agency Contract Laboratory Program Statement of Work, February 1988.

QUALITY CONTROL

Quality control procedures specified for these methods were followed. The following is a summary of the reporting forms.

Form	Information	Comments
2A	Surrogate Recovery	Meets all requirements.
3A	MS/MSD Recovery	Omitted, see note below.
4A	Method Blank Summary	Meets all requirements.
5A	Tune/ Mass Calibration	Meets all requirements.
6A	Initial Calibration Data	5 point calibration. Meets all requirements.
7A	Daily Calibration	Meets all requirements.
8A	Internal Standards	Meets all requirements.

NOTES:

- Holding time of the sample for analysis by GC/MS was exceeded due to extended analytical instrumentation difficulties.

- Spike and spike duplicate (MS and MSD) analysis were performed on the date of the sample analysis. However, due to lack of this sample, MS and MSD analyses were performed on a different sample. The matrix of the spiked sample is not truly representative of this sample. Therefore, form 3A is intentionally omitted so as not to be misleading.

DATA

The data and calibration files are archived on magnetic tape in lab 427 in the 325 building. The pertinent data files are listed on form 4A as "Lab File ID".

RESULTS

CLP Target Compounds: Acetone and 2-Butanone (MEK) were detected above the Contract Required Quantitation Limits. The analysis was performed on a diluted (1:5) sample for reasons described in the "Sample Preparation" section of this report. In the interest of minimizing GC/MS system degradation and completing the analysis, re-analysis of an un-diluted aliquot was not performed.

Form 1A for each sample lists the target compounds. The column "Q" on the Form 1A indicates the EPA defined data qualifier as defined on the next page:

"Q" Flag	Definition
U	Indicates compound was analyzed for but not detected.
J	Indicates an estimated value. Spectra meet criteria but response is below a quantifiable confidence level.
B	Indicates compound was found in the blank.
D	Indicates analysis was performed on a diluted sample.
X	Indicates a manually deleted result from the automated data system report. The mass spectra did not qualify upon review.
E	Indicates quantitation was performed outside of the established calibration range.

Tentatively Identified Compounds (TIC): Concentration estimates of TIC's are made by assuming that the response factors are one. Quantitation is then based on the nearest eluting internal standard. The results for the TIC compounds are shown on Form 1E for each sample. No TICs were found.

ANALYST [signature] for MM McBarney DATE 11-21-90 REVIEW [signature] DATE 11-21-90

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

BLANK

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: LAB BLANK

Sample wt/vol: 5 (g/mL) mL Lab File ID: >W1503

Level: (low/med) LOW Date Received: 11/15/90

% Moisture: not dec.---- Date Analyzed: 11/15/90

Column: (pack/cap) CAP Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L	Q	
74-87-3	Chloromethane	10.	IU	
74-83-9	Bromomethane	10.	IU	
75-01-4	Vinyl Chloride	10.	IU	
75-00-3	Chloroethane	10.	IU	
75-09-2	Methylene_Chloride	5.	IU	X
67-64-1	Acetone	3.	IJB	
75-15-0	Carbon Disulfide	5.	IU	X
75-35-4	1,1-Dichloroethene	5.	IU	
75-34-3	1,1-Dichloroethane	5.	IU	
540-59-0	1,2-Dichloroethene_(total)	5.	IU	
67-66-3	Chloroform	5.	IU	X
107-02-2	1,2-Dichloroethane	5.	IU	
78-93-3	2-Butanone	6.	IJB	
71-55-6	1,1,1-Trichloroethane	5.	IU	X
56-23-5	Carbon Tetrachloride	5.	IU	X
108-05-4	Vinyl Acetate	10.	IU	
75-27-4	Bromodichloromethane	5.	IU	
78-87-5	1,2-Dichloropropane	5.	IU	
10061-01-5	cis-1,3-Dichloropropene	5.	IU	
79-01-6	Trichloroethene	5.	IU	
124-48-1	Dibromochloromethane	5.	IU	X
79-00-5	1,1,2-Trichloroethane	5.	IU	
71-43-2	Benzene	5.	IU	X
10061-02-6	trans-1,3-Dichloropropene	5.	IU	
75-25-2	Bromoform	5.	IU	X
108-10-1	4-Methyl-2-pentanone	10.	IU	
591-78-6	2-Hexanone	10.	IU	X
127-18-4	Tetrachloroethene	5.	IU	X
79-34-5	1,1,2,2-Tetrachloroethane	5.	IU	X
108-88-3	Toluene	5.	IU	X
108-90-7	Chlorobenzene	5.	IU	X
100-41-4	Ethylbenzene	5.	IU	
100-42-5	Styrene	5.	IU	
133-02-7	Xylene (total)	5.	IU	

9 2 1 2 4 1 1 0 7 5

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

BLANK

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: LAB BLANK

Sample wt/vol: 5 (g/mL) mL Lab File ID: >W1503

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- Date Analyzed: 11/15/90

Column: CAP Dilution Factor: 1

Number TICs found: 0

CONCENTRATION UNITS:
(ug/L or ug/Kg) ug/L

	CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					
21.					
22.					
23.					
24.					
25.					
26.					
27.					
28.					
29.					
30.					

FORM I VOA-TIC 1/87 Rev.

1A
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

90-6940

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: 241-AN-106

Sample wt/vol: 5 (g/mL) mL Lab File ID: >W1508

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- Date Analyzed: 11/15/90

Column: (pack/cap) CAP Dilution Factor: 5

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L	Q
74-87-3	Chloromethane	50.	U D
74-83-9	Bromomethane	50.	U D
75-01-4	Vinyl Chloride	50.	U D
75-00-3	Chloroethane	50.	U D
75-09-2	Methylene_Chloride	5.	U X
67-64-1	Acetone	74.	BD
75-15-0	Carbon Disulfide	25.	U D
75-35-4	1,1-Dichloroethene	11.	J D
75-34-3	1,1-Dichloroethane	25.	U D
540-59-0	1,2-Dichloroethene_(total)	25.	U D
67-66-3	Chloroform	25.	U D
107-02-2	1,2-Dichloroethane	25.	U D
78-93-3	2-Butanone	75.	BD
71-55-6	1,1,1-Trichloroethane	5.	U X
56-23-5	Carbon Tetrachloride	25.	U D
108-05-4	Vinyl Acetate	50.	U D
75-27-4	Bromodichloromethane	25.	U D
78-87-5	1,2-Dichloropropane	25.	U D
10061-01-5	cis-1,3-Dichloropropene	25.	U D
79-01-6	Trichloroethene	22.	J D
124-48-1	Dibromochloromethane	25.	U D
79-00-5	1,1,2-Trichloroethane	25.	U D
71-43-2	Benzene	5.	U X
10061-02-6	trans-1,3-Dichloropropene	25.	U D
75-25-2	Bromoform	25.	U D
108-10-1	4-Methyl-2-pentanone	10.	U X
591-78-6	2-Hexanone	10.	U X
127-18-4	Tetrachloroethene	25.	U D
79-34-5	1,1,2,2-Tetrachloroethane	25.	U D
108-88-3	Toluene	22.	J D
108-90-7	Chlorobenzene	23.	J D
100-41-4	Ethylbenzene	25.	U D
100-42-5	Styrene	25.	U D
133-02-7	Xylene (total)	25.	U D

9 2 1 2 4 1 4 1 0 7 7

1E
VOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.
90-6940

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: 241-AN-106

Sample wt/vol: 5 (g/mL) mL Lab File ID: >W1508

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- Date Analyzed: 11/15/90

Column: CAP Dilution Factor: 5

Number TICs found: 0 CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

FORM I VOA-TIC 1/87 Rev.

2A

WATER VOLATILE SURROGATE RECOVERY

Lab Name:Battelle-PNL Contract:------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

	EPA SAMPLE NO.	S1 (TOL)#	S2 (BFB)#	S3 (DCE)#	OTHER	TOT OUT
01	LAB BLANK	93	94	91		0
02	90-6940	92	91	97		0
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

		QC LIMITS
S1 (TOL)	= Toluene-d8	(88-110)
S2 (BFB)	= Bromofluorobenzene	(86-115)
S3 (DCE)	= 1,2-Dichloroethane-d4	(76-114)

Column to be used to flag recovery values

* Values outside of contract required QC limits

D Surrogates diluted out

9 2 1 2 4 1 4 1 0 7 9

4A

VOLATILE METHOD BLANK SUMMARY

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Lab File ID: >W1503 Lab Sample ID: LAB BLANK

Date Analyzed: 11/15/90 Time Analyzed: 11:39

Matrix: (soil/water) WATER Level: (low/med) LOW

Instrument ID: WB46869

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01	90-6940	241-AN-106	>W1508	15:37
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS:

9 2 1 2 4 1 1 0 8 0

5A

VOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION - BROMOFLUOROBENZENE (BFB)

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Lab File ID: >W1501 BFB Injection Date: 11/15/90

Instrument ID: 70 2 BFB Injection Time: 10:28

Matrix: (soil/water) WATER Level: (low/med) LOW Column: (pack/cap) CAP

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% OF MASS 95	20.4
75	30.0 - 60.0% OF MASS 95	55.6
95	Base peak, 100% relative abundance	100.
96	5.0 - 9.0% of mass 95	6.7
173	Less than 2.0% of mass 174	0.0(0.0)1
174	Greater than 50.0% of mass 95	70.7
175	5.0 - 9.0% of mass 174	4.5(6.4)1
176	Greater than 95.0%, but less than 101.0% of mass 174	69.7(98.6)1
177	5.0 - 9.0% of mass 176	4.1(5.9)2

1-Value is % mass 174 2-Value is % mass 176

S TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01	CALIBRATION	DAILY CAL	>W1502	11/15/90	11:03
02	BLANK	LAB BLANK	>W1503	11/15/90	11:39
03	90-6940	241-AN-106	>W1508	11/15/90	15:37
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

page 1 of 1

FORM V VOA 1/87 Rev.

6A

VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name:Battelle-PNL Contract:------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Instrument ID: 70 2 Calibration Date(s):11/13/90 11/15/90

Matrix:(soil/water) WATER Level:(low/med) LOW Column:(pack/cap) CAP

Min $\overline{RRF}$ for SPCC(#) = 0.300 (0.250 for Bromoform) Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF20 =>W1306 RRF50 =>W1307
RRF100=>W1308 RRF150=>W1309 RRF200=>W1304

COMPOUND	RRF20	RRF50	RRF100	RRF150	RRF200	$\overline{RRF}$	% RSD
Chloromethane #	.409	.389	.411	.375	.366	.390	5.1#
Bromomethane	1.079	.999	1.063	.995	.985	1.024	4.2
Vinyl_Chloride *	.665	.610	.648	.600	.591	.623	5.1*
Chloroethane	.358	.346	.368	.343	.339	.351	3.4
Methylene_Chloride	.921	.853	.933	.880	.860	.890	4.0
Acetone	.180	.173	.140	.137	.165	.159	12.3
Carbon_Disulfide	2.366	2.127	2.310	2.176	2.186	2.233	4.5
1,1-Dichloroethene *	1.076	.936	1.022	.951	.954	.988	6.0*
1,1-Dichloroethane #	1.863	1.721	1.891	1.781	1.774	1.806	3.9#
1,2-Dichloroethene_(total)	1.121	1.000	1.082	1.013	1.018	1.047	5.0
loroform *	2.859	2.598	2.877	2.705	2.639	2.736	4.6*
1,2-Dichloroethane	1.967	1.773	2.000	1.874	1.808	1.884	5.2
2-Butanone	.081	.065	.062	.061	.058	.065	13.4
1,1,1-Trichloroethane	3.048	2.700	3.044	2.804	2.810	2.881	5.4
Carbon_Tetrachloride	2.961	2.646	3.051	2.806	2.757	2.844	5.7
Vinyl_Acetate	1.162	1.186	1.328	1.262	1.210	1.229	5.4
Bromodichloromethane	2.799	2.542	2.975	2.775	2.660	2.750	5.9
1,2-Dichloropropane *	.253	.228	.251	.230	.224	.237	5.8*
cis-1,3-Dichloropropene	.190	.167	.190	.175	.169	.178	6.4
Trichloroethene	.456	.403	.445	.394	.400	.419	6.8
Dibromochloromethane	.707	.637	.749	.675	.616	.677	7.9
1,1,2-Trichloroethane	.305	.267	.301	.273	.263	.282	7.0
Benzene	.638	.568	.618	.571	.568	.593	5.6
trans-1,3-Dichloropropene	.788	.722	.806	.731	.666	.743	7.5
Bromoform #	.590	.525	.619	.581	.534	.570	6.9#
4-Methyl-2-pentanone	.204	.171	.191	.181	.160	.181	9.4
2-Hexanone	.132	.122	.127	.120	.109	.122	7.0
Tetrachloroethene	.562	.466	.514	.466	.468	.495	8.6
1,1,2,2-Tetrachloroethane	.494	.417	.476	.442	.401	.446	8.8
Toluene *	.568	.498	.562	.511	.511	.530	6.1*
Chlorobenzene #	.864	.773	.851	.773	.689	.790	8.9#
Ethylbenzene *	.395	.340	.379	.348	.340	.360	7.0*
Styrene	.812	.720	.804	.722	.660	.744	8.6
Xylene_(total)	.469	.405	.449	.410	.399	.426	7.3
Toluene-d8	1.020	.895	.996	.898	.808	.923	9.3
Bromofluorobenzene	.828	.704	.779	.720	.651	.737	9.3
1,2-Dichloroethane-d4	1.732	1.560	1.723	1.634	1.584	1.647	4.8

7A

VOLATILE CONTINUING CALIBRATION CHECK

Lab Name:Battelle-PNL Contract:------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Instrument ID: 70 2 Calibration Date: 11/15/90 Time: 11:03

Lab File ID: >W1502 Init. Calib. Date(s):11/13/90 11/15/90

Matrix:(soil/water) WATER Level:(low/med) LOW Column:(pack/cap) CAP

Min RRF50 for SPCC(#) = 0.300 (0.250 for Bromoform) Max %D for CCC(*) = 25.0%

COMPOUND		RRF	RRF50	%D	
Chloromethane	#	.390	.410	5.2	#
Bromomethane		1.024	1.089	6.4	
Vinyl Chloride	*	.623	.640	2.8	*
Chloroethane		.351	.355	1.1	
Methylene_Chloride		.890	.926	4.2	
Acetone		.159	.241	51.3	
Carbon_Disulfide		2.233	2.268	1.6	
1,1-Dichloroethene	*	.988	1.022	3.4	*
1,1-Dichloroethane	#	1.806	1.881	4.1	#
1,2-Dichloroethene_(total)		1.047	1.085	3.6	
Chloroform	*	2.736	2.876	5.1	*
1,2-Dichloroethane		1.884	1.993	5.7	
2-Butanone		.065	.078	19.3	
1,1,1-Trichloroethane		2.881	3.101	7.6	
Carbon_Tetrachloride		2.844	3.036	6.8	
Vinyl Acetate		1.229	1.074	12.6	
Bromodichloromethane		2.750	2.884	4.9	
1,2-Dichloropropane	*	.237	.239	.8	*
cis-1,3-Dichloropropene		.178	.181	1.7	
Trichloroethene		.419	.442	5.4	
Dibromochloromethane		.677	.726	7.3	
1,1,2-Trichloroethane		.282	.293	4.0	
Benzene		.593	.613	3.5	
trans-1,3-Dichloropropene		.743	.775	4.3	
Bromoform	#	.570	.580	1.7	#
4-Methyl-2-pentanone		.181	.190	4.8	
2-Hexanone		.122	.138	13.2	
Tetrachloroethene		.495	.518	4.5	
1,1,2,2-Tetrachloroethane		.446	.462	3.5	
Toluene	*	.530	.548	3.4	*
Chlorobenzene	#	.790	.844	6.9	#
Ethylbenzene	*	.360	.378	4.8	*
Styrene		.744	.790	6.2	
Xylene_(total)		.426	.450	5.6	
Toluene-d8		.923	.980	6.1	
Bromofluorobenzene		.737	.775	5.2	
1,2-Dichloroethane-d4		1.647	1.828	11.0	

9 2 1 2 4 1 1 0 8 3

8A

VOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name:Battelle-PNL Contract:------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Lab File ID (Standard): >W1502 Date Analyzed:11/15/90

Instrument ID: 70 2 Time Analyzed: 11:03

Matrix:(soil/water) WATER Level:(low/med) LOW Column:(pack/cap) CAP

		IS1(BCM) AREA #	RT	IS2(DFB) AREA #	RT	IS3(CBZ) AREA #	RT
	12 HOUR STD	208474.	9.29	813860.	12.68	730326.	21.08
	UPPER LIMIT	416948.		1627720.		1460652.	
	LOWER LIMIT	104237.		406930.		365163.	
	EPA SAMPLE NO.						
01	LAB BLANK	216477.	9.31	853549.	12.72	760341.	21.09
02	90-6940	231620.	9.35	844668.	12.76	791340.	21.13
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS1 (BCM) = Bromochloromethane
IS2 (DFB) = 1,4-Difluorobenzene
IS3 (CBZ) = Chlorobenzene-d5

UPPER LIMIT = + 100%
of internal stansard area.
LOWER LIMIT = - 50%
of internal standard area.

Column used to flag internal standard area values with an asterisk

FORM VIII VOA 1/87 Rev.

9 2 1 2 4 1 4 1 0 9 4

ORGANIC COMPOUND ANALYSIS REPORT

SEMIVOLATILE COMPOUNDS

SAMPLE ANALYSIS REPORTED

Analysis of one sample for semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS) is the subject of this report.

SAMPLE PREPARATION

The sample and the method blank were extracted in the 325 building Shielded Analytical Laboratory (SAL) according to the procedure in PNL-ALO-120 with some differences. Only 54 mL sample was available so matrix spike analyses could not be performed. Consequently, only 100 mL distilled water was carried through the method blank analysis. After the liquid sample was decanted into a separatory funnel, the solids were washed with distilled water and the wash water was decanted into the separatory funnel. Methylene chloride was used to extract the organics from this aqueous phase (pH adjusted to 12). When the sample was acidified there was a vigorous evolution of gas. The acidified sample was again extracted with solvent. The organic solutions underwent final concentration in Lab 302.

ANALYSIS METHOD

The procedure followed for these GC/MS analyses was PNL-ALO-345. The HP-5890/5970 GC/MS (WB38473) used for the analyses is in Lab 702 of the 325 building.

QUALITY CONTROL

The QC procedures in the analytical procedure were followed. The following lists the attached CLP forms that relate to QC and summarizes the QC results.

Form	Information	Comments
2D	Surrogate Recovery	The surrogate recoveries for the blank met requirements. Recoveries for the acid surrogates were zero.
3D	MS/MSD Recovery	Not included.
4B	Method Blank Summary	The blank had higher than usual phthalate concentrations.
5B	Tune/Mass Calibration	Met requirements.
6B,C	Initial Calibration	Met requirements.

9 2 1 2 4 1 4 1 0 8 5

7B,C	Daily Calibration	Met requirements.
8B,C	Internal Standards	Met requirements.

The following is a discussion of deviations from QC and protocol requirements.

- Holding time. The holding time for extracting the sample was exceeded because of instrumentation problems. The holding time for analysis after extraction was not exceeded.
- Surrogate recoveries. The acid surrogates were probably lost because of reactions with matrix components. This has been shown in past analyses of waste solutions in which nitrated derivatives of the surrogates were found. Potential derivatives of the surrogates found in this sample were nitrophenol and dinitrophenol and possibly the phenoxy- compounds shown in the report for the tentatively identified compounds, Form 1F.
- MS/MSD recoveries. Insufficient sample to perform these analyses.
- Blank analyses. Phthalate concentrations were too high probably because of contamination from plastics in the hot cells.

DATA

The data and calibration are archived on magnetic tape in the 325 building 702 laboratory. The following is the list of the pertinent files.

File Name	Sample Number	Sample Analyzed
>K1901		DFTPP tune/mass calibration check
>K1902		Daily calibration
>K1903		Method blank
>K1905	90-6940	

RESULTS

CLP Target Compounds: As seen in the attached 1B,C Forms, target compounds found are Phenol (100 ppb), Nitrobenzene (35 ppb), 2-nitrophenol (110 ppb), Benzoic acid (620 ppb), 2,4-dinitrophenol, (1100 ppb), and the same phthalates that are in the blank. The following defines the Q-flags in the Form 1's

"Q" Flag	Definition
U	Indicates the compound was analyzed for but not detected, the U-flagged concentration is the Contract Required Quantitation Limit

J	Indicates an estimated value, spectra meet criteria but response is below Contract Required Quantitation Limit
B	Indicates compound was found in the blank
X	Indicates compound was manually deleted because all requirements were not met
D	Indicates analysis was performed on a diluted sample
E	Indicates quantitation outside the calibration range

Tentatively Identified Compounds (TIC): The attached 1F Forms show results for the TIC's. Concentration estimates for the TIC's are made assuming that the response factor for each TIC is one. Then the peak area for each TIC is compared to the area of the nearest internal standard (for which concentrations are known) to estimate the TIC concentrations. Identification of the TIC is made by a computer search of the NIST mass spectral library to attempt a match with the spectrum of each TIC's. The TIC's reported as "Unknown" did not have satisfactory matches with library spectra.

ANALYST [signature] DATE 11-21-90 REVIEW [signature] DATE 11-21-90

This page is intentionally left blank

1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

BLANK

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: METHOD BLK

Sample wt/vol: 100 (g/mL) mL Lab File ID: >K1903

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- dec. ---- Date Extracted: 10/26/90

Extraction: (Sepf/Cont/Sonc) SEPF Date Analyzed: 11/19/90

GPC Cleanup: (Y/N) N pH: Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L	Q
108-95-2	Phenol	100.	U
111-44-4	bis(2-Chloroethyl)Ether	100.	U
95-57-8	2-Chlorophenol	100.	U
541-73-1	1,3-Dichlorobenzene	100.	U
106-46-7	1,4-Dichlorobenzene	100.	U
100-51-6	Benzyl alcohol	100.	U
95-50-1	1,2-Dichlorobenzene	100.	U
95-48-7	2-Methylphenol	100.	U
39638-32-9	bis(2-chloroisopropyl)ether	100.	U
106-44-5	4-Methylphenol	100.	U
621-64-7	N-Nitroso-Di-n-propylamine	100.	U
67-72-1	Hexachloroethane	100.	U
98-95-3	Nitrobenzene	100.	U
78-59-1	Isophorone	100.	U
88-75-5	2-Nitrophenol	100.	U
105-67-9	2,4-Dimethylphenol	100.	U
65-85-0	Benzoic acid	500.	U
111-91-1	bis(2-Chloroethoxy)methane	100.	U
120-83-2	2,4-Dichlorophenol	100.	U
120-82-1	1,2,4-Trichlorobenzene	100.	U
91-20-3	Naphthalene	100.	U
106-47-8	4-Chloroaniline	100.	U
87-68-3	Hexachlorobutadiene	100.	U
59-50-7	4-Chloro-3-methylphenol	100.	U
91-57-6	2-Methylnaphthalene	100.	U
77-47-4	Hexachlorocyclopentadiene	100.	U
88-06-2	2,4,6-Trichlorophenol	100.	U
95-95-4	2,4,5-Trichlorophenol	500.	U
91-58-7	2-Chloronaphthalene	100.	U
88-74-4	2-Nitroaniline	500.	U
131-11-3	Dimethylphthalate	100.	U
208-96-8	Acenaphthylene	100.	U
606-20-2	2,6-Dinitrotoluene	100.	U

1C
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

BLANK

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: METHOD BLK

Sample wt/vol: 100 (g/mL) mL Lab File ID: >K1903

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- dec. ---- Date Extracted: 10/26/90

Extraction: (Sepf/Cont/Sonc) SEPF Date Analyzed: 11/19/90

GPC Cleanup: (Y/N) N pH: Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L	Q
99-09-2	3-Nitroaniline	500.	U
83-32-9	Acenaphthene	100.	U
51-28-5	2,4-Dinitrophenol	500.	U
100-02-7	4-Nitrophenol	500.	U
132-64-9	Dibenzofuran	100.	U
121-14-2	2,4-Dinitrotoluene	100.	U
84-66-2	Diethylphthalate	9.	J
7005-72-3	4-Chlorophenyl-phenylether	100.	U
86-73-7	Fluorene	100.	U
100-01-6	4-Nitroaniline	500.	U
534-52-1	4,6-Dinitro-2-methylphenol	500.	U
86-30-6	N-Nitrosodiphenylamine (1)	100.	U
101-55-3	4-Bromophenyl-phenylether	100.	U
118-74-1	Hexachlorobenzene	100.	U
87-86-5	Pentachlorophenol	500.	U
85-01-8	Phenanthrene	100.	U
120-12-7	Anthracene	100.	U
84-74-2	Di-n-butylphthalate	210.	
206-44-0	Fluoranthene	100.	U
129-00-0	Pyrene	100.	U
85-68-7	Butylbenzylphthalate	28.	J
91-94-1	3,3'-Dichlorobenzidine	200.	U
56-55-3	Benzo(a)anthracene	100.	U
218-01-9	Chrysene	100.	U
117-81-7	bis(2-Ethylhexyl)phthalate	190.	
117-84-0	Di-n-octylphthalate	100.	U
205-99-2	Benzo(b)fluoranthene	100.	U
207-08-9	Benzo(k)fluoranthene	100.	U
50-32-8	Benzo(a)pyrene	100.	U
193-39-5	Indeno(1,2,3-cd)pyrene	100.	U
53-70-3	Dibenz(a,h)anthracene	100.	U
191-24-2	Benzo(g,h,i)perylene	100.	U

(1) - Cannot be separated from Diphenylamine

9 2 1 2 4 1 4 1 0 9 0

1F

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

BLANK

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: METHOD BLK

Sample wt/vol: 100 (g/mL) mL Lab File ID: >K1903

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- dec. ---- Date Extracted: 10/26/90

Extraction: (Sepf/Cont/Sonc) SEPF Date Analyzed: 11/19/90

GPC Cleanup: (Y/N) N pH: 7 Dilution Factor: 1

Number TICs found: 6 CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unknown	4.07	270.	J
2.	Unknown	4.82	110.	J
3.	Unknown	5.12	140.	J
4.	Unknown	5.96	610.	J
5.	Unknown	6.40	120.	J
6.	Unknown	20.02	500.	J
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

FORM I SV-TIC 1/87 Rev.

1B

EPA SAMPLE NO.

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

90-6940

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: 241-AN-106

Sample wt/vol: 54 (g/mL) mL Lab File ID: >K1905

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- dec. ---- Date Extracted: 10/26/90

Extraction: (Sepf/Cont/Sonc) SEPF Date Analyzed: 11/19/90

GPC Cleanup: (Y/N) N pH: 11 Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L	Q
108-95-2	Phenol	100.	J
111-44-4	bis(2-Chloroethyl)Ether	190.	U
95-57-8	2-Chlorophenol	190.	U
541-73-1	1,3-Dichlorobenzene	190.	U
106-46-7	1,4-Dichlorobenzene	190.	U
100-51-6	Benzyl alcohol	190.	U
95-50-1	1,2-Dichlorobenzene	190.	U
95-48-7	2-Methylphenol	190.	U
39638-32-9	bis(2-chloroisopropyl)ether	190.	U
106-44-5	4-Methylphenol	190.	U
621-64-7	N-Nitroso-Di-n-propylamine	190.	U
67-72-1	Hexachloroethane	190.	U
98-95-3	Nitrobenzene	35.	J
78-59-1	Isophorone	190.	U
88-75-5	2-Nitrophenol	110.	J
105-67-9	2,4-Dimethylphenol	190.	U
65-85-0	Benzoic acid	620.	J
111-91-1	bis(2-Chloroethoxy)methane	190.	U
120-83-2	2,4-Dichlorophenol	190.	U
120-82-1	1,2,4-Trichlorobenzene	190.	U
91-20-3	Naphthalene	190.	U
106-47-8	4-Chloroaniline	190.	U
87-68-3	Hexachlorobutadiene	190.	U
59-50-7	4-Chloro-3-methylphenol	190.	U
91-57-6	2-Methylnaphthalene	190.	U
77-47-4	Hexachlorocyclopentadiene	190.	U
88-06-2	2,4,6-Trichlorophenol	190.	U
95-95-4	2,4,5-Trichlorophenol	930.	U
91-58-7	2-Chloronaphthalene	190.	U
88-74-4	2-Nitroaniline	930.	U
131-11-3	Dimethylphthalate	190.	U
208-96-8	Acenaphthylene	190.	U
606-20-2	2,6-Dinitrotoluene	190.	U

9 2 1 2 4 1 4 1 0 9 2

1C
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.
90-6940

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: 241-AN-106

Sample wt/vol: 54 (g/mL) mL Lab File ID: >K1905

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- dec. ---- Date Extracted: 10/26/90

Extraction: (Sepf/Cont/Sonc) SEPF Date Analyzed: 11/19/90

GPC Cleanup: (Y/N) N pH: 11 Dilution Factor: 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L	Q
99-09-2	3-Nitroaniline	930.	U
83-32-9	Acenaphthene	190.	U
51-28-5	2,4-Dinitrophenol	1100.	
100-02-7	4-Nitrophenol	930.	U
132-64-9	Dibenzofuran	190.	U
121-14-2	2,4-Dinitrotoluene	190.	U
84-66-2	Diethylphthalate	20.	JB
7005-72-3	4-Chlorophenyl-phenylether	190.	U
86-73-7	Fluorene	190.	U
100-01-6	4-Nitroaniline	930.	U
534-52-1	4,6-Dinitro-2-methylphenol	930.	U
86-30-6	N-Nitrosodiphenylamine (1)	190.	U
101-55-3	4-Bromophenyl-phenylether	190.	U
118-74-1	Hexachlorobenzene	190.	U
87-86-5	Pentachlorophenol	930.	U
85-01-8	Phenanthrene	190.	U
120-12-7	Anthracene	190.	U
84-74-2	Di-n-butylphthalate	380.	B
206-44-0	Fluoranthene	190.	U
129-00-0	Pyrene	190.	U
85-68-7	Butylbenzylphthalate	120.	JB
91-94-1	3,3'-Dichlorobenzidine	370.	U
56-55-3	Benzo(a)anthracene	190.	U
218-01-9	Chrysene	190.	U
117-81-7	bis(2-Ethylhexyl)phthalate	46.	JB
117-84-0	Di-n-octylphthalate	190.	U
205-99-2	Benzo(b)fluoranthene	190.	U
207-08-9	Benzo(k)fluoranthene	190.	U
50-32-8	Benzo(a)pyrene	190.	U
193-39-5	Indeno(1,2,3-cd)pyrene	190.	U
53-70-3	Dibenz(a,h)anthracene	190.	U
191-24-2	Benzo(g,h,i)perylene	190.	U

(1) - Cannot be separated from Diphenylamine

1F

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

90-6940

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Matrix: (soil/water) WATER Lab Sample ID: 241-AN-106

Sample wt/vol: 54 (g/mL) mL Lab File ID: >K1905

Level: (low/med) LOW Date Received: 08/31/90

% Moisture: not dec.---- dec. ---- Date Extracted: 10/26/90

Extraction: (Sepf/Cont/Sonc) SEPF Date Analyzed: 11/19/90

GPC Cleanup: (Y/N) N pH: 11 Dilution Factor: 1

Number TICs found: 30

CONCENTRATION UNITS: (ug/L or ug/Kg) ug/L

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.	Unknown	4.12	530.	J
2.	Unknown	4.39	660.	J
3.	Unknown	4.61	290.	J
4.	Unknown	4.73	330.	J
5.	Unknown	5.66	700.	J
6.	Unknown	5.99	500.	J
7. 822877	Cyclohexanone, 2-chloro-	6.35	370.	J
8. 822866	Cyclohexane, 1,2-dichloro-,	6.58	1200.	J
9.	Unknown	6.94	310.	J
10.	Unknown	7.35	830.	J
11. 51422754	Cyclohexane, 1-bromo-2-chlor	7.97	1100.	J
12.	Unknown	9.27	6400.	J
13. 122996	Ethanol, 2-phenoxy-	9.35	710.	J
14.	Unknown	9.97	13000.	J
15.	Unknown	10.05	270.	J
16.	Unknown	10.49	600.	J
17.	Unknown	10.55	620.	J
18. 16736428	2,6-Octadiene, 2,7-dimethyl-	14.66	410.	J
19. 104687	Ethanol, 2-(2-phenoxyethoxy)	16.52	590.	J
20.	Unknown	17.93	230.	J
21.	Unknown	18.50	150.	J
22.	Unknown	20.16	3000.	J
23.	Unknown	23.24	290.	J
24. 7204162	Ethanol, 2-[2-(2-phenoxyetho	23.73	1200.	J
25.	Unknown	25.79	360.	J
26.	Unknown	29.05	1200.	J
27.	Unknown	29.38	1500.	J
28.	Unknown	30.73	350.	J
29.	Substituted phenoxy compound	33.24	1100.	J
30.	Unknown	36.34	320.	J

2C
WATER SEMIVOLATILE SURROGATE RECOVERY

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	OTHER	TOT OUT
01	BLANK	73	70	82	53	67	87		0
02	90-6940	88	85	85	0 *	0 *	0 *		3
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

		QC LIMITS
S1 (NBZ)	= Nitrobenzene-d5	(35-114)
S2 (FBP)	= 2-Fluorobiphenyl	(43-116)
S3 (TPH)	= Terphenyl-d14	(33-141)
S4 (PHL)	= Phenol-d5	(10-94)
S5 (2FP)	= 2-Fluorophenol	(21-100)
S6 (TBP)	= 2,4,6-Tribromophenol	(10-123)

Column to be used to flag recovery values
* Values outside of contract required QC limits
D Surrogates diluted out

9 2 1 2 4 1 4 1 0 9 5

4B
SEMIVOLATILE METHOD BLANK SUMMARY

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Lab File ID: >K1903 Lab Sample ID: METHOD BLK

Date Extracted 10/26/90 Extraction: (SepF/Cont/Sonc) SEPF

Date Analyzed: 11/19/90 Time Analyzed: 17:39

Matrix: (soil/water) WATER Level: (low/med) LOW

Instrument ID: 70 1

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
01	90-6940	241-AN-106	>K1905	11/19/90
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS:

9 2 1 2 4 1 4 1 0 9 6

5B

SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS CALIBRATION - DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab Name:Battelle-PNL Contract:| the contra

Lab Code: | your EP Case No.: | the CaSAS No.: | the S SDG No.: | the S

Lab File ID: >K1901 DFTPP Injection Date:11/19/90

Instrument ID: 70 1 DFTPP Injection Time: 14:23

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	33.4
68	Less than 2.0% of mass 69	0.0(0.0)1
69	Mass 69 relative abundance	47.
70	Less than 2.0% of mass 69	0.0(0.0)1
127	40.0 - 60.0% of mass 198	44.9
197	Less than 1.0% of mass 198	0.0
198	Base Peak, 100% relative abundance	100.
199	5.0 - 9.0% of mass 198	6.6
275	10.0 - 30.0% of mass 198	22.6
365	Greater than 1.00% of mass 198	1.21
441	Present, but less than mass 443	8.1
442	Greater than 40.0% of mass 198	58.7
443	17.0 - 23.0% of mass 442	11.0(18.8)2

1-Value is % mass 69 2-Value is % mass 442

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01	DAILY CALIB	DAILY CALIB	>K1902	11/19/90	15:24
02	BLANK	METHOD BLK	>K1903	11/19/90	17:39
03	90-6940	241-AN-106	>K1905	11/19/90	19:41
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

page 1 of 1

FORM V SV 1/87 Rev.

6B

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Instrument ID: 70 1 Calibration Date(s): 11/19/90 11/90/90

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF20 =>K1606 RRF50 =>K1607
RRF80 =>K1608 RRF120=>K1609 RRF160=>K1610

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Phenol *	1.972	1.798	1.881	1.792	1.725	1.834	5.2*
bis(2-Chloroethyl)ether	1.784	1.748	1.886	1.963	2.089	1.894	7.3
2-Chlorophenol	1.517	1.438	1.539	1.556	1.529	1.516	3.0
1,3-Dichlorobenzene	1.458	1.346	1.408	1.464	1.408	1.417	3.4
1,4-Dichlorobenzene *	1.694	1.587	1.736	1.621	1.607	1.649	3.8*
Benzyl alcohol	.950	.896	.994	.963	.903	.941	4.4
1,2-Dichlorobenzene	1.475	1.370	1.491	1.438	1.336	1.422	4.7
2-Methylphenol	1.270	1.215	1.307	1.233	1.163	1.238	4.4
bis(2-chloroisopropyl)ether	1.488	1.454	1.491	1.379	1.267	1.415	6.7
4-Methylphenol	1.318	1.232	1.344	1.367	1.341	1.320	4.0
N-Nitroso-di-n-propylamine #	.739	.696	.735	.646	.602	.684	8.7#
Hexachloroethane	.684	.655	.711	.663	.651	.673	3.7
Nitrobenzene	.400	.378	.386	.368	.354	.377	4.7
Isophorone	.792	.754	.774	.782	.748	.770	2.4
2-Nitrophenol *	.227	.219	.240	.240	.240	.233	4.2*
2,4-Dimethylphenol	.326	.300	.313	.304	.306	.310	3.3
Benzoic acid	0.000	.221	.245	.253	.248	.242	5.8
bis(2-Chloroethoxy)methane	.553	.513	.519	.508	.502	.519	3.9
2,4-Dichlorophenol *	.315	.304	.306	.312	.297	.307	2.3*
1,2,4-Trichlorobenzene	.323	.299	.308	.305	.305	.308	2.9
Naphthalene	1.088	.995	.996	.946	.911	.987	6.8
4-Chloroaniline	.357	.420	.448	.431	.442	.420	8.7
Hexachlorobutadiene *	.137	.131	.141	.137	.134	.136	2.6*
4-Chloro-3-methylphenol *	.320	.305	.320	.307	.295	.309	3.3*
2-Methylnaphthalene	.656	.611	.622	.598	.593	.616	4.0
Hexachlorocyclopentadiene #	.183	.222	.241	.260	.263	.234	13.9#
2,4,6-Trichlorophenol *	.416	.391	.408	.391	.387	.399	3.1*
2,4,5-Trichlorophenol	.432	.407	.434	.420	.415	.422	2.7
2-Chloronaphthalene	1.270	1.185	1.214	1.159	1.128	1.191	4.6
2-Nitroaniline	0.000	.433	.432	.410	.397	.418	4.1
Dimethylphthalate	1.513	1.429	1.464	1.445	1.447	1.460	2.2
Acenaphthylene	2.055	1.942	1.931	1.905	1.856	1.938	3.8
2,6-Dinitrotoluene	.358	.354	.369	.366	.362	.362	1.7
3-Nitroaniline	0.000	.328	.422	.409	.391	.388	10.7
Acenaphthene *	1.293	1.193	1.184	1.132	1.097	1.180	6.3*
2,4-Dinitrophenol #	0.000	.128	.172	.198	.220	.180	21.9#
4-Nitrophenol #	0.000	.154	.166	.167	.143	.158	7.3#

6C

SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Instrument ID: 70 1 Calibration Date(s): 11/19/90 11/90/90

Min $\overline{RRF}$ for SPCC(#) = 0.050 Max %RSD for CCC(*) = 30.0%

LAB FILE ID: RRF20 =>K1606 RRF50 =>K1607
RRF80 =>K1608 RRF120=>K1609 RRF160=>K1610

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Dibenzofuran	1.714	1.638	1.673	1.642	1.594	1.652	2.7
2,4-Dinitrotoluene	.456	.472	.501	.490	.487	.481	3.7
Diethylphthalate	1.595	1.528	1.599	1.564	1.549	1.567	1.9
4-Chlorophenyl-phenylether	.571	.561	.571	.557	.552	.562	1.5
Fluorene	1.299	1.247	1.288	1.242	1.214	1.258	2.8
4-Nitroaniline	0.000	.234	.306	.346	.370	.314	18.9
4,6-Dinitro-2-methylphenol	0.000	.128	.152	.159	.166	.151	10.9
N-Nitrosodiphenylamine (1) *	.556	.508	.531	.500	.486	.516	5.3*
4-Bromophenyl-phenylether	.180	.176	.181	.178	.176	.178	1.4
Hexachlorobenzene	.192	.177	.188	.182	.184	.185	3.2
Pentachlorophenol *	0.000	.094	.110	.121	.128	.113	13.2*
Phenanthrene	1.145	1.088	1.103	1.091	1.060	1.098	2.8
Anthracene	1.093	1.039	1.058	1.030	1.010	1.046	3.0
Di-n-butylphthalate	1.771	1.744	1.773	1.723	1.691	1.740	2.0
Fluoranthene *	1.141	1.089	1.123	1.106	1.112	1.114	1.7*
Pyrene	1.531	1.446	1.441	1.409	1.366	1.438	4.2
Butylbenzylphthalate	1.064	1.001	1.003	.968	.946	.997	4.5
3,3'-Dichlorobenzidine	.237	.212	.187	.235	.263	.227	12.7
Benzo(a)anthracene	1.147	1.091	1.143	1.099	1.123	1.121	2.2
Chrysene	1.198	1.077	1.092	1.064	1.015	1.089	6.2
bis(2-Ethylhexyl)phthalate	1.543	1.428	1.460	1.374	1.279	1.417	7.0
Di-n-octylphthalate *	2.906	2.659	2.984	2.665	2.307	2.704	9.8*
Benzo(b)fluoranthene	1.257	1.163	1.345	1.344	1.382	1.298	6.8
Benzo(k)fluoranthene	1.151	1.082	1.147	.943	.941	1.053	10.0
Benzo(a)pyrene *	1.058	1.021	1.144	1.122	1.066	1.082	4.6*
Indeno(1,2,3-cd)pyrene	.872	.915	1.030	1.175	1.142	1.027	13.0
Dibenz(a,h)anthracene	.829	.827	.946	.929	.931	.892	6.6
Benzo(g,h,i)perylene	.888	.899	.999	.977	.968	.946	5.2
Nitrobenzene-d5	.392	.377	.393	.386	.374	.385	2.3
2-Fluorobiphenyl	1.401	1.280	1.307	1.243	1.215	1.289	5.6
Terphenyl-d14	.843	.810	.861	.834	.820	.834	2.4
Phenol-d5	1.646	1.577	1.651	1.611	1.509	1.599	3.7
2-Fluorophenol	1.349	1.324	1.433	1.403	1.371	1.376	3.1
2,4,6-Tribromophenol	.073	.074	.081	.079	.083	.078	5.7

(1) Cannot be separated from Diphenylamine

7B
SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name:Battelle-PNL Contract:------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Instrument ID: 70 1 Calibration Date: 11/19/90 Time: 15:24

Lab File ID: >K1902 Init. Calib. Date(s):11/19/90 11/90/90

Min RRF50 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.0%

COMPOUND		$\overline{RRF}$	RRF50	%D	
Phenol	*	1.834	1.820	.7	*
bis(2-Chloroethyl)ether		1.894	1.872	1.2	
2-Chlorophenol		1.516	1.441	4.9	
1,3-Dichlorobenzene		1.417	1.424	.5	
1,4-Dichlorobenzene	*	1.649	1.654	.3	*
Benzyl alcohol		.941	.934	.8	
1,2-Dichlorobenzene		1.422	1.470	3.4	
2-Methylphenol		1.238	1.230	.6	
bis(2-chloroisopropyl)ether		1.415	1.447	2.2	
4-Methylphenol		1.320	1.297	1.7	
N-Nitroso-di-n-propylamine	#	.684	.532	22.1	#
Hexachloroethane		.673	.683	1.6	
Nitrobenzene		.377	.370	1.8	
Isophorone		.770	.758	1.6	
2-Nitrophenol	*	.233	.230	1.4	*
2,4-Dimethylphenol		.310	.302	2.6	
Benzoic acid		.242	.222	8.3	
bis(2-Chloroethoxy)methane		.519	.505	2.7	
2,4-Dichlorophenol	*	.307	.309	.5	*
1,2,4-Trichlorobenzene		.308	.309	.5	
Naphthalene		.987	.985	.2	
4-Chloroaniline		.420	.343	18.2	
Hexachlorobutadiene	*	.136	.141	3.7	*
4-Chloro-3-methylphenol	*	.309	.314	1.3	*
2-Methylnaphthalene		.616	.637	3.5	
Hexachlorocyclopentadiene	#	.234	.186	20.3	#
2,4,6-Trichlorophenol	*	.399	.402	.8	*
2,4,5-Trichlorophenol		.422	.422	.2	
2-Chloronaphthalene		1.191	1.180	.9	
2-Nitroaniline		.418	.420	.4	
Dimethylphthalate		1.460	1.477	1.2	
Acenaphthylene		1.938	1.891	2.4	
2,6-Dinitrotoluene		.362	.367	1.6	
3-Nitroaniline		.388	.209	46.2	
Acenaphthene	*	1.180	1.171	.8	*
2,4-Dinitrophenol	#	.180	.133	26.1	#
4-Nitrophenol	#	.158	.156	1.3	#

7C

SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Instrument ID: 70 1 Calibration Date: 11/19/90 Time: 15:24

Lab File ID: >K1902 Init. Calib. Date(s): 11/19/90 11/90/90

Min RRF50 for SPCC(#) = 0.050 Max %D for CCC(*) = 25.0%

COMPOUND		$\overline{RRF}$	RRF50	%D	
Dibenzofuran		1.652	1.636	1.0	
2,4-Dinitrotoluene		.481	.493	2.5	
Diethylphthalate		1.567	1.585	1.1	
4-Chlorophenyl-phenylether		.562	.582	3.4	
Fluorene		1.258	1.233	2.0	
4-Nitroaniline		.314	.222	29.2	
4,6-Dinitro-2-methylphenol		.151	.128	15.1	
N-Nitrosodiphenylamine (1)	*	.516	.500	3.1	*
4-Bromophenyl-phenylether		.178	.175	1.8	
Hexachlorobenzene		.185	.180	2.7	
Pentachlorophenol	*	.113	.100	11.4	*
Phenanthrene		1.098	1.083	1.3	
Anthracene		1.046	1.037	.9	
Di-n-butylphthalate		1.740	1.787	2.7	
Fluoranthene	*	1.114	1.138	2.1	*
Pyrene		1.438	1.413	1.7	
Butylbenzylphthalate		.997	1.023	2.7	
3,3'-Dichlorobenzidine		.227	.198	12.8	
Benzo(a)anthracene		1.121	1.139	1.6	
Chrysene		1.089	1.115	2.3	
bis(2-Ethylhexyl)phthalate		1.417	1.498	5.7	
Di-n-octylphthalate	*	2.704	3.000	10.9	*
Benzo(b)fluoranthene		1.298	1.331	2.5	
Benzo(k)fluoranthene		1.053	1.128	7.2	
Benzo(a)pyrene	*	1.082	1.094	1.1	*
Indeno(1,2,3-cd)pyrene		1.027	.979	4.7	
Dibenz(a,h)anthracene		.892	.881	1.3	
Benzo(g,h,i)perylene		.946	.950	.4	
Nitrobenzene-d5		.385	.379	1.5	
2-Fluorobiphenyl		1.289	1.282	.6	
Terphenyl-d14		.834	.853	2.3	
Phenol-d6		0.000	0.000	0.0	
2-Fluorophenol		1.376	1.275	7.3	
2,4,6-Tribromophenol		.078	.070	10.0	

(1) Cannot be separated from Diphenylamine

8B

SEMIVOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Lab File ID (Standard): >K1902 Date Analyzed: 11/19/90

Instrument ID: 70 1 Time Analyzed: 15:24

		IS1(DCB) AREA #	RT	IS2(NPT) AREA #	RT	IS3(ANT) AREA #	RT
	12 HOUR STD	44809.	5.76	167024.	8.64	83281.	15.68
	UPPER LIMIT	89618.		334048.		166562.	
	LOWER LIMIT	22404.		83512.		41640.	
	EPA SAMPLE NO.						
01	BLANK	46590.	5.74	157887.	8.61	79925.	15.67
02	90-6940	47200.	5.75	167303.	8.64	76985.	15.69
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS1 (DCB) = 1,4-Dichlorobenzene-d4
IS2 (NPT) = Naphthalene-d8
IS3 (ANT) = Acenaphthene-d8

UPPER LIMIT = + 100% of internal stansard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

9 2 1 2 4 1 1 0 2

8C

SEMIVOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: Battelle-PNL Contract: ------

Lab Code: ------ Case No.: ------ SAS No.: ------ SDG No.: ------

Lab File ID (Standard): >K1902 Date Analyzed: 11/19/90

Instrument ID: 70 1 Time Analyzed: 15:24

		IS4(PHN) AREA #	RT	IS5(CRY) AREA #	RT	IS3(PRY) AREA #	RT
	12 HOUR STD	138138.	23.08	109972.	34.44	94591.	38.84
	UPPER LIMIT	276276.		219944.		189182.	
	LOWER LIMIT	69069.		54986.		47295.	
	EPA SAMPLE NO.						
01	BLANK	126558.	23.06	95509.	34.41	78536.	38.80
02	90-6940	129666.	23.08	102383.	34.43	77724.	38.82
03							
04							
05							
06							
07							
08							
09							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							

IS4 (PHN) = Phenanthrene-d10
IS5 (CRY) = Chrysene-d12
IS6 (PRY) = Perylene-d12

UPPER LIMIT = + 100% of internal stansard area.
LOWER LIMIT = - 50% of internal standard area.

Column used to flag internal standard area values with an asterisk

page 1 of 1

FORM VIII SV-2 1/87 Rev.

9 2 1 2 4 1 1 0 3

This page is intentionally left blank

9 1 1 2 4 1 4 1 1 0 5

#8

Action Item 1-29-91:1

J. A. Voogd
Westinghouse Hanford Company
Richland, Washington

Grout Treatment Facility Unit Managers' Meeting
March 1991

Action Item 1-29-91:1

WHC and DOE will provide greater detail on the practical quantitation limits and the relationship with the contract laboratory procedures by the next Unit Managers Meeting.

Basis

On January 29, 1991, Westinghouse provided results from organic chemical analyses conducted upon wastes withdrawn from Tank 241-AN-106. These wastes are planned to constitute the waste feed to the first Grout Treatment Facility mixed waste disposal action.

Discussion developed surrounding the effect of laboratory dilution of sample extracts for volatile organic analyses. In particular, a contrast between published quantitation limit values and values applied in these results were questioned.

What is a PQL?

A "Practical Quantitation Limit (PQL) is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions."[1]

How is a PQL applied?

PQLs are applied specific to the method of analysis. Method 8240 of SW-846 "Gas Chromatography/Mass Spectrometry for Volatile Organics" states:

> 1.3 The practical quantitation limit (PQL) of Method 8240 for an individual compound is approximately 5 μg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 μg/L for ground water (see Table 2). PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

---

1 SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition, Revision 1, December 1987 (p. ONE - 9).

What is the value of a PQL?

Table 2 of SW-846, Method 8240, lists groundwater and low soil/sediment PQL values for 35 organic consitituents, e.g.:

TABLE 2. PRACTICAL QUANTITATION LIMITS (PQL) FOR VOLATILE ORGANICS[a]

			Practical Quantitation Limit[b]	
			Ground Water	Low Soil/Sediment
		CAS Number	µg/L	µg/Kg
1.	Chloromethane	74-87-3	10	10
5.	Methylene Chloride	75-09-2	5	5
6.	Acetone	67-64-1	100	100
13.	2-Butanone	78-93-3	100	100
14.	1,1,1-Trichloroethane	71-55-6	5	5

"a Sample PQLs are highly matrix dependant. The PQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependant PQLs.

b PQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, PQLs will be higher based on the % moisture in each sample."

How is a PQL increased for other matrices?

For samples which are not ground water, the PQL for a constituent is multiplied by a specified factor for that other sample type. This is due to the increasing interferance and complexity of other components in the sample which change the accuracy and precision (the basis of a PQL) of measurement.

From SW-846, Method 8240:

Other Matrices:	Factor[1]
Water miscible liquid waste	50
High-level soil & sludges	125
Non-water miscible waste	500

1 PQL = [PQL for groundwater (Table 2)] X [Factor]. For non-aqueous samples, the factor is on a wet-weight basis.

What is a CRQL and how does it compare to a PQL?

A Contract Required Quantitation Limit (CRQL) is effectively a negotiated PQL between a client and the laboratory based upon the achievable limit of measurement for that laboratory. They are thereafter treated as "laboratory specific PQLs"

The CRQLs between Westinghouse Hanford Company and Battelle Pacific Northwest Laboratories for SW-846 Method 8240 analysis are generally at or near the PQLs of SW-846.

Are the CRQLs used in these analyses any better or more representative than the PQLs?

YES. Although PQLs are multiplied up to 500 times for different matrices CRQLs are only multiplied by dilution factors - in this case, five.

As an example, the corrected PQL for acetone is 5,000 μg/L, while the corrected CRQL for acetone is 50 μg/L.

SUMMARY

- The quantitation limits applied to these analyses were considerably lower than PQLs for water miscible liquid waste and our analyses more stringent than that required by SW-846.

Laboratory Quantitation Limit Comparisons For Analysis of 241-AN-106 Organic Analytes[1]						
Volatile Analyte	PQL Groundwater Analysis (SW-846)	CRQL[2] Water Analysis	PQL Water Miscible Waste	CRQL Diluted Waste Analysis[3]	Reported Value	Hit[4]
Chloromethane	10	10	500	50	50	U[5]
Bromomethane	10	10	500	50	50	U
Vinyl Chloride	10	10	500	50	50	U
Chloroethane	10	10	500	50	50	U
Methylene Chloride	5	10	250	50	5	U
Acetone	100	10	5,000	50	74	X
Carbon Disulfide	5	10	250	50	25	U
1,1-Dichloroethene	5	10	250	50	11	J
1,1-Dichloroethane	5	10	250	50	25	U
trans-1,2-Dichloroethene	5	*[6]	250	*	25	U
Chloroform	5	10	250	50	25	U
1,2-Dichloroethane	5	10	250	50	25	U
2-Butanone	100	10	5,000	50	75	X
1,1,1-Trichloroethane	5	10	250	50	5	U
Carbon Tetrachloride	5	10	250	50	25	U
Vinyl Acetate	50	*	2,500	*	50	U
Bromodichloromethane	5	10	250	50	25	U

Laboratory Quantitation Limit Comparisons For Analysis of 241-AN-106 Organic Analytes[1]						
Volatile Analyte	PQL Groundwater Analysis (SW-846)	CRQL[2] Water Analysis	PQL Water Miscible Waste	CRQL Diluted Waste Analysis[3]	Reported Value	Hit[4]
1,1,2,2-Tetrachloroethane	5	10	250	50	25	U
1,2-Dichloropropane	5	10	250	50	25	U
trans-1,3-Dichloropropene	5	10	250	50	25	U
Benzene	5	10	250	50	5	U
cis-1,3-Dichloropropene	5	10	250	50	25	U
Bromoform	5	10	250	50	25	U
2-Hexanone	50	10	2,500	50	10	U
4-Methyl-2-pentanone	50	10	2,500	50	10	U
Tetrachloroethene	5	10	250	50	25	U

1. All units reported in µg/L.

2. Contract Required Quantitation Limit listed in Contract Laboratory Program (CLP) statement of work.

3. For a dilution factor of five.

4. Reported value above CRQL.

5. U = Analysis conducted, but compound not detected.
   J = Analyte detected, but below quantitation limit.
   X = Analyte detected above quantitation limit.

6. No QL listed.

#9

CORRESPONDENCE DISTRIBUTION COVERSHEET

Author	Addressee	Correspondence No.
R. F. Wood	E. A. Bracken	9152593

Subject: GROUT MONTHLY STATUS REPORT FOR WASHINGTON STATE DEPARTMENT OF ECOLOGY

INTERNAL DISTRIBUTION

Approval	Date	Name	Location	w/att
		Correspondence Control	R1-03	X
		President's Office	B3-01	X
		R. J. Bliss	R3-04	X
		J. S. Hill	H4-57	X
		D. J. Newland	R2-28	X
		S. M. Price	H4-57	X
		J. H. Roecker	R2-28	X
		J. A. Voogd	R4-03	X
		G. F. Williamson	R4-01	X
X	4/24/91	R. F. Wood	R4-01	X
		GWJ File/LB	R4-01	X
		GT File/LB	R4-03	X

Westinghouse
Hanford Company

P.O. Box 1970 Richland, WA 99352

April 24, 1991 9152593

Ms. E. A. Bracken, Director
Environmental Restoration Division
U.S. Department of Energy
Richland Operations Office
Richland, Washington 99352

Dear Ms. Bracken:

GROUT MONTHLY STATUS REPORT FOR WASHINGTON STATE DEPARTMENT OF ECOLOGY

Attached is the Grout Monthly Status Report for March. This report is to be included in the Unit Managers Meeting minutes as agreed with Washington State Department of Ecology. Please include the attachment into the March 22 meeting minutes.

Very truly yours,

G. W. Jackson, Manager
Grout Facilities
Defense Waste Remediation Division

bce

Attachments 1

DOE-RL - K. W. Bracken
C. E. Clark
J. L. Daily
R. O. Puthoff (w/o attachment)
G. S. Rokkan
S&WE - J. D. King

GROUT MONTHLY STATUS REPORT FOR WASHINGTON STATE DEPARTMENT OF ECOLOGY

- Grout Vaults 102-105 - Construction activities for Project B-714, Grout Vaults 102-105 performed this month included concrete placement for the final wall lift on Vault 102 and the third wall lift for Vault 103. Presentations to U.S. Environmental Protection Agency (EPA) and Washington State Department of Ecology (Ecology) concerning a delay in Hanford Federal Facility Agreement and Consent Order (Tri-Party Agreement) milestones by 19 months have been conducted.

- Grout Disposal Vaults 106-109 - The Kaiser Engineers Hanford Company (KEH) has submitted a revised schedule and cost estimate for the Project W-125, Grout Disposal Vaults 106-109. The schedule and estimate are based on the 19 month delay to the original Tri-Party Agreement milestone for the vaults and is being used as the cost and schedule basis to support validation of the project.

- Portable Instrument Houses - The Westinghouse Hanford Company (WHC) Purchasing continues their efforts to transfer responsibility of the Project W-118, Portable Instrument Houses contract from U.S. Ecology to NOVA Technologies. The current schedule forecasts completion of the transfer during April 1991. Design completion for this effort is also forecasted for April.

- Grout Vault Ventilation System - The KEH has extended the bid due date for the design, fabrication and delivery of the Project W-089, Grout Vault Ventilation System by three weeks. The extension to April 10 was required to allow additional vendors to prepare bids.

- Spare 104/102-AP In-Tank Mixer Pump - The Procurement Specification for the 104/102-AP Submersible In-Tank Mixer Pump was issued. The Procurement Specification was revised to incorporate comments and outstanding amendments. Procurement efforts will start upon approval of a Capital Equipment Not Related to Construction funded Cost Account Authorization and Cost Account Plan. The purchase of another spare In-Tank Mixer Pump replaces the existing spare that is being withdrawn from inventory for use in tank 104-AP. A sole source procurement is being considered because of the manufacturer's unique design, construction capabilities and patents on the mixer pump.

- Vault Support Equipment - In support of Project W-213, Vault Support Equipment, a demonstration of the Gould pump which has been specified as the Excess Water Pump and the Short Term Leachate Pump took place on February 26 at the vendor's facility. The purpose of this test was to determine the lowest level of fluid that this pump can be expected to remove. The pump was able to remove fluid to a level of approximately one-half inch, which far exceeded the maximum of three inches. Design

requirements for Grout Vault Excess Water and Leachate Pumps was sent to KEH so that an evaluation could be performed detailing the impacts these requirements will have on the existing designs.

- Tank AP-104 Upgrades - The Preliminary Safety Analysis Report (PSAR) for Project W-062, Tank AP-104 Upgrades is being written, with Pacific Northwest Laboratories (PNL) providing support for the bulk of the work. This effort was expected to be complete by the end of March. However, due to higher priority work within the safety group, the report is not expected to be out for review until late April. Construction cannot begin until the PSAR is approved. The expense funded jumper package is going through final WHC sign-off in preparation for release and fabrication. Comments on the definitive design are being resolved.

- Grout Final Safety Analysis Report (FSAR) - A meeting with the Waste Management Safety & Environmental Advisory Council (SEAC) subcouncil was held to discuss the Operational Safety Requirements (OSRs) for Chapter 11 of FSAR.

  Efforts continue to review and disposition the SEAC subcouncil comments for Chapters 5 and 6. Work continues on to evaluating and closing open items from the Safety Analysis Open Items list for Chapters 5 and 6.

  Review of the Revision A of the Grout FSAR by the SEAC, Tank Waste Disposal (TWD) Subcouncil continues. The review is forecast to be completed on schedule by March 31, 1991. Subcouncil issues are being addressed on an "as needed" basis by the FSAR team. During March three information meetings were provided at the SEAC TWD Subcouncil's request.

- Grout Treatment Facility Dangerous Waste Permit Application (Part B) - The Part B Notice of Deficiency response table has been reviewed by the Grout Technology group and comments forwarded to the Environmental Division for incorporation into the final response table. The final response table is due to Ecology on April 12.

- Land Disposal Restrictions - A letter has been drafted requesting the EPA concurrence with the DOE-RL/WHC's determination of treatment standards necessary for grout feeds and grouted waste. The letter identifies the treatment standards as:

  - Nonwastewater for F001-F005 constituents of the grout feed
  - Wastewater for non F001-F005 constituents of the grout feed
  - Nonwastewater for grouted waste

The EPA has given a preliminary indication of concurrence with this interpretation; however, the issue must be closed to assure the Hanford Grout Disposal Program (HGDP) can proceed with grouting of tank 106-AN waste material.

- Grout Formulation Task Force - The work conducted here will provide the necessary planning to support the grout formulation activities.

  Input on grout performance criteria and test methods for demonstrating compliance with those criteria was prepared for inclusion in the Grout Formulation Criteria Document. A plan for developing and verifying grout formulations for 106-AN and double-shell slurry feed wastes was developed by a team composed of WHC, Oak Ridge National Laboratory, and PNL staff.

- Characterization - WHC-SD-TP-065, Revision 0, "Tank 241-AN-106 Characterization Results" was issued as an externally released document on March 8.

- Liner Irradiation - A letter report describing experimental work to evaluate the resistance of Nokorode 705M to the effects of gamma irradiation and the chemicals likely to contact it was provided to WHC for review. As described in the report, the evaluation did not reveal any obvious deficiencies of the material that would disqualify it for the intended use. An informal presentation on the results of this work was given to WHC on February 22 and at the Unit Managers Meeting on March 22, 1991.

- Phosphate/Sulfate Waste (PSW) Core Sampling - A meeting was held with Process Engineering, Nuclear Safety, and Safety Analysis Report Development to address safety documentation for PSW vault core sampling As a result of this meeting and subsequent management discussion, the safety documentation will be incorporated into the PSW Hazards Identification and Evaluation (SD-WM-SAR-027).

- Integrated Tank Waste Core Sampling Schedule - A response to the draft "Integrated Tank Waste Core Sampling Schedule" was transmitted to Waste Management Technology. The draft schedule does not support HGDP coring requirements. The Ecology has required that Vault 102 be cored prior to pouring Vault 103. A two year lead time is estimated for procuring a dedicated grout coring truck. This will lead to additional schedule slippage unless a tank farm core truck can be used.

Distribution:

R.M. Carosino	DOE-RL (A4-52)
C.E. Clark	DOE-RL (A6-95)
L.P. Diediker	WHC (T1-30)
D.L. Duncan	EPA (HW-074)
C.J. Geier	WHC (H4-57)
D.W. Hendrickson	WHC (R4-03)
J.S. Hill	WHC (H4-57)
R.D. Izatt	DOE-RL (A6-95)
G.W. Jackson	WHC (R4-01)
D.A. Jones	WHC (H4-16)
J.D. King	SWEC (A4-35)
K.S. McCullough	WHC (N1-83)
T.M. Michelena	Ecology
L.L. Powers	WHC (B2-35)
S.M. Price	WHC (H4-57)
G.S. Rokkan	DOE-RL (A5-21)
J.E. Van Beek	WHC (R3-27)
T.B. Veneziano	WHC (B2-35)
J.A. Voogd	WHC (R4-03)
J.L. Waite	WHC (B2-35)
B.D. Williamson	WHC (B3-15)
G.F. Williamson	WHC (R4-01)
J.J. Witczak	Ecology
T.J. Woebkenberg	SWEC (A4-35)
G.D. Wright	WHC (R3-09)

ADMINISTRATIVE RECORD (Grout Treatment Facility, TD-2-1)
[Care of Susan Wray, WHC (H4-22C)]

Washington State Department of Ecology, Nuclear And Mixed Waste Library,
Mail Stop PV-11

Environmental Protection Agency, Region 10, HW-074