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7. Abstract

This technical project plan addresses the Processing and Analytical Laboratories support to the Grout Treatment Facility in response to the following test plans and statement of work: (1) WHC-SD-WM-TP-130 for tank 241-AP-105, (2) WHC-SD-WM-TP-117 for tank 241-AP-106, (3) WHC-SD-WM-TP-136, and (4) statement of work WHC-SOW-93-0005.

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LIST OF TERMS

AA	atomic absorption
ANSI	American National Standards Institute
AO	Analytical Operations
B&A	before and after
CLP	EPA Contract Laboratory Program
CVAA	cold vapor atomic absorption
DL	detection limit
DQO	data quality objective
DUP	duplicate
ECN	Engineering Change Notice
EPA	Environmental Protection Agency
EP-TOX	extraction procedure toxicity
GEA	gamma energy analysis
GTF	Grout Treatment Facility
HASM	Hanford Analytical Services Management
IC	ion chromatography
ICP/AES	inductively coupled plasma/atomic emission spectroscopy
LCCS	Laboratory Customer Computer System
LMCS	Laboratory Measurement Control System
NIST	National Institute & Standard Technology
PAL	Processing and Analytical Laboratories
PNL	Battelle Pacific Northwest Laboratories
PSC	Process Support Chemist
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
QC	quality control
RPD	relative percent difference
RSA	Request for Special Analysis
SOW	statement of work
SPL	sample
SPK	spike
SW-846	test methods for evaluating solid waste
TCLP	toxic characteristic leach procedure
TFO	Tank Farm Operations
TOC	total organic carbon
TOX	total organic halide
TPP	technical project plan
TWRS	Tank Waste Remediation System
VOA	volatile organic analysis

1.0 INTRODUCTION

This technical project plan (TPP) addresses the Processing and Analytical Laboratories (PAL) support to the Grout Treatment Facility (GTF) in response to the following three test plans: (1) "Tank 241-AP-105 Sampling and Characterization Plan," WHC-SD-WM-TP-130, Revision 0, (2) "Tank 241-AP-106 Sampling and Characterization Plan," WHC-SD-WM-TP-117, Revision 0, and (3) "Hanford Grout Disposal Program Campaign 102 Characterization and Test Plan," WHC-SD-WM-TP-136, Revision 0. This TPP focuses on identifying areas of these plans and associated statement of work (SOW) (WHC-SOW-93-0005) where the laboratory may (1) take exception to the requirements, (2) require further clarification of the requirements, or (3) recommend alternatives to the plan or SOW.

1.1 ASSUMPTIONS

The budgets, schedules, and deliverables described in this TPP are based on the assumptions that (1) supporting documents are approved and provided in a timely manner, (2) samples will be delivered as planned and (3) that the priority in sampling and analysis for the GTF will not be superseded by other Tank Waste Remediation System (TWRS) programs. Meeting the requirements of this work also assumes that funding will be in place to support the activity and that no unplanned changes in waste disposal requirements, staffing, or equipment availability will occur.

1.2 CUSTOMER RESPONSIBILITIES

The customer must clearly identify data and analytical measurements that will be used for regulatory compliance such as delisting, land disposal restrictions and other applications where "mandatory" procedure compliance (40 FR 4440) is required. This information is needed to ensure that the laboratory utilizes the proper procedures and quality control (QC) and prepares the proper documentation. Based on the laboratory interpretation of the GTF characterization plans, rigorous procedure compliance is required for TCLP and organic testing for tank 102-AP and possibly the organic analyses for tanks 105-AP and 106-AP.

Tank Farm Operations (TFO) is responsible for notifying the laboratory at least 24 hours in advance of the expected sample shipment. If samples are going to be delivered after 3:00 P.M., the laboratory should be notified by 12:00 P.M. so that proper shift operations can be planned. Tank Farm Operations is also responsible for shipping samples with properly completed chain of custody forms, seals, identification tags, and shipping papers. Samples must be taken in a manner that will minimize impact to sample integrity and will not create hazardous conditions for laboratory personnel. Sampling to control quality of sampling activities, such as field blanks, are the responsibility of TFO.

Unused samples shall be maintained for a period of at least 60 days after the receipt of the data reports by Grout Technology. If the storage capacity of the laboratory facility is exceeded or safe efficient operating conditions are impacted, Grout Technology will be notified of the need for

early disposal. Disposal of samples are tracked by the Laboratory Customer Computer System (LCCS) and are not tracked using the original chain of custody form.

2.0 WORKSCOPE AND ADMINISTRATION

2.1 PROJECT DESCRIPTION

Waste in tank 102-AP has been blended and is designated as the next feed to the GTF. Before the waste in this tank can be grouted it must be characterized to confirm: (1) that the chemical and radiological components are within the grout acceptance criteria (Hendrickson 1991) and (2) that the grout formulation will produce a grout with acceptable physical and leaching properties. Toxic characteristic leach procedure and American National Standards Institute (ANSI) 16.1 leaching tests will be performed on grout specimens made from this waste. Because this is the last characterization of the waste before disposal, a Resource Conservation and Recovery Act (RCRA) data package to support the results will be prepared.

Waste in tank 105-AP is a candidate for grouting in a future campaign. This characterization plan does not contain any grout specimen preparation or leaching tests. The primary objective of the characterization is to determine if the chemical and radiological components are within the grout acceptance criteria and to plan the necessary blending and formulation requirements for the future GTF feed tanks. Because the organic content in tank 105-AP may be required to be shown to be acceptable before blending, these analyses will be reported in a Environmental Protection Agency Contract Laboratory Program (CLP) type data package.

Waste in tank 106-AP is a dilute waste candidate for blending with waste from 101-AW or 105-AP to produce a feed for the GTF. This plan includes characterization and testing to evaluate the grout formulation and grout properties. This plan is similar to that for 102-AP--but not as extensive because it is not the final feed to GTF. Because the organic content in tank 106-AP may be required to be shown to be acceptable before blending, these analyses will be reported in a CLP type data package.

The analytical and testing requirements for the three tanks are summarized in Appendix A. The number of individual samples, composites, grout specimens, and leach tests for the three tanks are provided in the last page of Appendix A. Because these requirements have been modified recently, Grout Technology should review these tables to verify that they accurately reflect the test plans and revisions.

2.2 ADMINISTRATION

2.2.1 Organization and Personnel

The PAL organization is presently undergoing a reorganization and additional changes may occur during the course of this characterization. The key personnel involved in performing GTF characterization at the 222-S Laboratory is outlined in Appendix B. Appendix B has two parts: (1) a general laboratory organization chart of the key organizations involved in the characterization and (2) a table of key personnel. Analytical Operations (AO) is responsible for maintaining the facility, sample control, providing technologist and chemist support to projects and disposal of waste generated

in the process. If a project requires special process tests and treatment before being analyzed by the analytical laboratory, these tests and characterization subsampling will be performed by a process support chemist (PSC) in the Process Chemistry Laboratory. In these cases, the PSC will act as the program manager for the overall laboratory project and provide the single point of contact for the customer. If the characterization project does not require special testing or preparation, the samples will be subsampled by AO and a project coordinator from the Project Support group will be identified as the single point of contact. Even if a PSC is the single point of contact for a project, AO will normally identify a project coordinator to work with the PSC to coordinate the analytical activities. The AO is responsible for providing the project coordination support between the customer or the PSC and the Analytical Laboratory. They are also responsible for providing the technical (analytical chemists) support in each of the specific technological areas. Laboratory Scheduling and Work Control are responsible for preparing, statusing, and maintaining the detailed schedules for completing the analytical work from each tank and providing data package assembly support. Laboratory Data Management is responsible for final assembly, pagination, and coordination of error correction, prior to transmitting data to Hanford Analytical Services Management (HASM). The Laboratory Data Quality organization is responsible for development of quality assurance (QA) and QC records, performing internal QA/QC audits, and QA/QC reviews of the data package. The Process Laboratory and Technology statistics team will provide data analysis and evaluation to determine sampling requirements and waste homogeneity. The function of the HASM organization is to provide an interface to the program and the laboratory through the SOW and to perform the final validation of the data package. This organization is also responsible for long term planning of laboratory schedules and budgets and for preparing cost variance reports for the work performed. Environmental Administrative Records was responsible for copying and distributing the data packages. This organization and its responsibilities have been transferred to Information Resource Management. Information Resource Management is also responsible for long term storage and maintenance of the records.

2.2.2 Project Coordination

The grout PSC, or their designee, is the single point of contact for the 222-S Laboratory for this program during the normal business hours (7:00 A.M. to 4:30 P.M.). Alternate points of contact are the grout analytical project coordinator and the AO Manager. During off shifts the shift manager is the emergency point of contact. The PSC is responsible for communicating program requirements to the Analytical Laboratory through the analytical project coordinator. The project coordinator is responsible for coordinating, compiling, reviewing and reporting work done by the AO. The project coordinator is also responsible for assessing the status (percent complete) of the analyses and providing this information to the Laboratory Scheduling and Work Control organization for reporting. Data package narratives and summaries are prepared by the project coordinator. The project coordinator is responsible for the final analytical technical review of the data package. Additional review and evaluation of data to determine: (1) that the waste samples are representative, (2) that the waste meets the necessary grouting criteria, and (3) that the grout formulation and product quality are satisfactory will be performed by the PSC, Grout Technology and the Process

Laboratory and Technology statistics team. These data evaluations may be reported independently from the analytical data package.

Changes or proposed changes in the Analytical Laboratory Operations that impact the test plans or SOW are communicated to the PSC, Grout Technology and HASM by phone, status reports or during routine meetings depending on the significance and urgency of the change requirement. Changes may be documented in laboratory notebooks, internal characterization change notices for changes that do not impact schedule or scope or by engineering change notices (ECNs) to the base documents. Characterization change notices may be prepared by anyone working on the project; however, it is the responsibility of the project coordinator to collect, track, and report their contents. The purpose of these change notices is to document the characterization activities as accurately as possible. These change notices will become a part of the final data package.

2.2.3 Equipment

The equipment used in an analysis is identified on the analytical batch sheet in the data package. In case of extended equipment failure at 222-S Laboratory, Battelle Pacific Northwest Laboratories (PNL) would be considered for meeting the analysis requirements. A formal routine maintenance schedule is not used for most instrumentation. Most instrument components are electrical (not mechanical) and do not require routine maintenance. Some small routine maintenance tasks are included in the analytical procedures when appropriate. Instruments are normally operated until their performance indicates there is a problem. Maintenance will then be performed by (1) the appropriate chemist, (2) laboratory instrument personnel or, (3) the instrument manufacturer (depending on the nature and source of the problem).

2.2.4 Procedures

The analytical procedures used for this program have been identified in the appendices of each test plan. A more complete list of 222-S Laboratory procedures, applicable to this program, is provided in Appendix C of this TPP. Because the test plans for 105-AP and 106-AP are older, the procedures in Appendix C may differ from those found in the plan. The latest version will be used. Hanford Analytical Services Management will be notified when significant revisions are made in the procedures during the course of characterization.

Procedures are prepared and controlled in accordance with Section 5.1 "Analytical Procedures" of the "Laboratories Administration Manual," WHC-CM-5-4. After changes have been incorporated and approved, new controlled copies of the procedure are distributed. If procedure changes must be made on specific samples because of matrix effects or other uncontrollable conditions, the change will be documented on the batch summary narrative or in the master narrative of the data package.

3.0 TECHNICAL AND ANALYTICAL ABILITY

3.1 SAMPLE RECEIVING AND SHIPPING

Samples are received into the laboratory by the sample custodian or their designee in accordance with procedure LO-090-101. On off shift, the shift manager is the designated sample custodian. The sample custodian verifies the condition of the samples received, signs the chain of custody, logs the sample into the laboratory, and stores the sample in a secured area according to established procedures.

Samples that must be sent out of the 222-S Laboratory for analysis will be shipped in accordance with the proper shipping procedures and will be accompanied by a chain of custody record.

3.2 SAMPLE HANDLING AND STORAGE

Waste samples from the tanks are stored in shielded transport casks (pigs) or in the 222-S Laboratory shielded sample storage facility at ambient temperature until they are transferred to the hot cell for subsampling and testing. Samples are kept sealed when not in use to minimize evaporation and loss of volatile components.

Wastes from the GTF tanks are not expected to contain significant quantities of solids. Homogenization is performed by simple magnetic stirring systems. Occasionally the salt concentrations of the waste are so high that the solutions become saturated and precipitate when removed from the warmer tank environment. In these cases it may be necessary to warm the samples to 40-60ºC to resolubilize the salts and to make a small preliminary (1:1) dilution in water to stabilize the solution before subsampling. If small quantities of solids are present in the samples, the PSC will decide how the subsamples will be taken. Subsamples could be taken while stirring or solids could be separated and analyzed separately. This solids treatment activity will be performed either by the PSC in the hot cell or by the Hot Cell and Sample Preparation group in the Analytical Laboratory.

Because sample preparations (acid, water, and fusion digestions) are now being performed by the Analytical Laboratory rather than the PSC, Sections 7.1.1 to 7.1.4 of the 105-AP and 106-AP are no longer applicable. Homogeneous liquids will be acid digested for inductively coupled plasma (ICP) metals analysis and diluted in water for anion characterization. The digestion methods used at 222-S Laboratory are based on SW-846 3050 or 3010 procedures for solids and liquids respectively and not the CLP SOW-787 Method C. If dilutions are required, they will be made in acid or water depending on the analyte as identified in Appendix A. Any digestions of solids will be done using the procedures identified in Appendix B.

Liquid subsamples and solids, if present and analyzed separately, are taken in the hot cell and submitted to the 222-S Laboratory for specific analyses based on a "Request for Special Analysis" (RSA) form. The RSAs are prepared by the PSC and communicated to the analytical project coordinator and laboratory leader. Preparations and analyses are then tracked and statused

using the LCCS. Subsamples and their preparations are stored and tracked by the sample custodian.

Because of the high radioactive nature of the samples, they cannot be preserved and stored in strict accordance with SW-846 requirements. Wastes are sampled, transported, homogenized, subsampled, and stored in a manner that provides the best sample integrity possible within the limitations of the containment systems needed to provide radiological protection to personnel.

The quantities of sample and the time period that samples can be archived is limited. Retention and disposal of samples and solutions will be made in accordance with laboratory standard operating procedures. When storage space becomes full or affects routine operations, the program will be requested to identify samples for disposal.

3.3 ANALYTICAL METHODS

All three test plans (Section 1.0) identify the procedures in the GTF Part B permit (DOE-RL 1992) as the procedures to be used for GTF waste characterization. In addition, the plans identify applicable procedures in their appendices (106-AP (Appendix D), 105-AP and 102-AP (Appendix C). The procedures listed in the test plans are more extensive than those identified in the Part B permit.

Procedures are listed in Table 3-13 of the Part B permit and described in Section 3.6 of that document. This section also identifies some potential problems that are expected in applying these procedures to Hanford wastes. The procedures identified for metals are either SW-846 inductively coupled plasma/atomic emission spectroscopy (ICP/AES) or atomic absorption (AA) methods. The 102-AP test plan does not identify all the metals identified in the Part B permit. The laboratory will report the analytes identified in the more recent test plans rather than the list in the Part B permit. The procedures identified in the test plan appendices and in Appendix B of this TPP for metals are 222-S Laboratory procedures that are based on the SW-846 methods. The mercury cold vapor method described in the Part B permit and test plans has been changed and no longer uses a gold amalgam concentrator. These methods are based on the same technology but may not follow all analytical and QC steps exactly.

Procedure modifications may be incorporated to accommodate (1) the complex composition of Hanford waste, (2) the radioactive nature of the samples, or (3) the data quality objectives (DQOs) of the program in the most cost effective manner. It is the laboratory's understanding that "mandatory" SW-846 procedures are only required for certain regulatory activities such as delisting and toxic characteristic testing (40 FR 4440). The SW-846 procedures are designed to provide results that are needed to "prove the negative" or that a result is below a specified regulatory value.

When some of these procedures and data rejection (QC) criteria, intended for soils and waters, are applied to chemically diverse and complex highly radioactive wastes they will result in an unnecessary impact to the cost and schedule of the program. For example, if the blank (no specific elements specified) for the ICP is outside the normal control limits, the entire batch of samples would be rerun under strict adherence to the SW-846 ICP procedure.

However, when analyzing high salt waste matrices which contains over 200,000 ug/g of sodium, it is not unusual to obtain a higher than normal sodium blank that is still less than 1% of the sodium in the sample. Because low level sodium analyses are obviously not important to the characterization, the SW-846 QC for this procedure is not explicitly followed.

Based on the laboratory's understanding of the test plans, the laboratory procedures are expected to meet the objectives of the program. However, it is important for the customer to also evaluate the detection limits, performance criteria, and data provided to determine if the DQOs for the program are being met. If the proposed procedures and results are inadequate, the laboratory should be notified of the deficiencies so that corrective actions can be taken.

The Part B permit identifies the Environmental Protection Agency (EPA) ion chromatograph (IC) method 300.0 for analysis of nitrate, nitrite, chloride, fluoride, sulfate and phosphate. This method is primarily designed for waters. The IC method used by the 222-S Laboratory uses the same technology but may not follow the EPA method exactly for the same reasons identified above for metals. The laboratory may also use a spectrophotometric method (LA-645-001) for nitrite that is identified in the test plan appendices. If present in significant quantities, water soluble organics such as acetate and formate may bias IC fluoride results high.

SW-846 methods for hydroxide, total organic carbon (TOC), and total organic halide (TOX) are also identified in the Part B permit. The SW-846 method for hydroxide (9040) is a pH determination procedure. The method used by the laboratory (LA-661-102) for highly alkaline wastes is a potentiometric titration with a standard acid with barium chloride added to remove carbonate and phosphate interferences. The TOC method used is based on the furnace oxidation of organics with coulometric titration of the generated carbon dioxide. The technology for determining TOC varies depending on the commercial instrumentation used. This method may not follow SW-846 procedures precisely for reasons already identified. None of the test plans include TOX as a required analysis as described in the Part B Permit; however, more extensive organic analyses are planned and therefore TOX will not be performed.

The test plans for 105-AP and 106-AP in Section 5.5, "Criteria/Constraints," indicate that "equivalent" procedures to SW-846 and EPA 300.0 for IC will be used when applicable. The laboratory has not done the extensive testing required to demonstrate that its modified methods are technically "equivalent" to the EPA procedures. It is the laboratories understanding that equivalency is only important when "mandatory" SW-846 procedures are required as discussed in 40 FR 4440. The laboratory believes that the QC (calibrations, standards, duplicate, spikes and blanks) applied provides the performance criteria needed to meet the Grout program objectives.

The Part B permit and the test plans identify 222-S Laboratory radiochemical methods for waste characterization. The methods identified are correct except for the tritium (H-3) method (LA-218-211) which has recently been changed(LA-218-214) to utilize disposable microdistillation technology in place of glass distillation equipment. No methods exist at the 222-S Laboratory for determining the Pu-241 and Am-242 isotopes identified in the QAPjP for tank 102-AP. The program may want to consider calculating these

isotopes using the analysis of Pu-239/240 and Am-241 and the reactor code estimates of the isotopic ratios after correcting for Pu-241 decay. It may be possible to determine these isotopes by mass spectrometry at PNL if their concentrations are high enough.

The characterization plans for 105-AP and 106-AP, Sections 5.4.2.2 identify SW-846 organic methods for characterizing the wastes. The 102-AP test plan has identified the EPA CLP organic methods as being applicable. If the candidate tanks (105-AP, 106-AP) are analyzed for organics at the 222-S Laboratory, the procedures will be based on CLP methods and not SW-846. Application of regulatory organic methods to the high nitrate/nitrite salt waste has created some analytical problems. Nitration of organic surrogates has produced low surrogate recoveries that can impact interpretation and use of the data. Limited sample quantities and relatively large sample size requirements needed to meet desired detection levels have prevented full compliance with some QC requirements. Presently the organic analyses for all the tanks are planned to be done at PNL.

The TCLP has replaced the extraction procedure toxicity procedure identified in the Part B permit. The procedure used at Hanford is scaled down by a factor of ten. It uses a 10 g sample size in place of 100 g and 200 mL of extraction fluid in place of the 2000 mL specified in the SW-846 1311 procedure. The regional EPA has agreed that the reduced sample size is not significant as long as measurements at the TCLP regulatory limits can be achieved. The point (step) in the procedure that spikes are added to the TCLP extract is also being discussed with the regional EPA. If this modification is not approved, the spikes may be added at both points to evaluate this potential problem of applying the TCLP to high salt and alkaline waste forms. If the TCLP analyte can be shown to be less than 20 times the TCLP limit in the original sample, the TCLP test will not be required for that analyte.

4.0 QUALITY ASSURANCE AND QUALITY CONTROL

Description and general direction of the laboratory QC and QA program is found in the "Quality Assurance Program Plan for the Laboratory Analysis of Environmental Samples," WHC-SD-CP-QAPP-003 and the "Quality Assurance Project Plan for the Analysis of Highly Radioactive Mixed Waste Samples in Support of Environmental Activities on the Hanford Site," WHC-SD-CP-QAPP-002. New revisions for both these documents are in the draft form and should be approve in Fiscal Year 1993. The requirements in these documents are secondary to the QA/QC requirements in the test plans or grout project specific QA project plan (QAPjP).

The QC applied to each sample is summarized in the tables in Appendix A. The instrument or method QC is found in the analytical procedures.

4.1 CALIBRATIONS

The calibration procedures and requirements are defined in each analytical procedure. Frequency of calibration depends on the technique. Some methods--such as ICP--are calibrated daily and verified after running a specified number of samples. Other methods--such as uranium by fluorimeter--use internal standards and are calibrated with each analysis. Some methods which use highly stable instrumental systems--such as counting equipment and spectrophotometers--are calibrated only when daily check standards or routine laboratory control standards show that the system calibration has changed.

Calibration records are included in data packages such as that requested for 102-AP. The test plans for the 105-AP and 106-AP (Section 5.4) request that calibration dates be reported and that calibration data be available on demand. Calibration dates are not normally requested. The ability of the laboratory to recall calibration information at a later date may be difficult or impossible. Some calibration records are stored on instrumentation computer systems whose software or hardware are occasionally updated making retrieval of old records difficult. Based on the response (Review Comment Record) from the grout program for this TPP, data packages will not be prepared for 105-AP and 106-AP tanks and calibration dates do not need to be included with the data.

4.2 STANDARDS

The original test plans (Section 5.4.2.1 for 105-AP, and Section 7 and 10.2 of the QAPjP for 102-AP) request at least two Laboratory Measurement Control Standards (LMCS) or a "reference sample" before and after (B&A) each batch of samples. If the purpose of these standards are to ensure the control of the method during the running of the samples, bracketing the samples with standards may be inappropriate and excessive depending on the method. This requirement has recently been modified in an ECN to the test plans requiring two standards only for those methods where it is normally required for instrument control.

Methods such as ICP and AA use calibration verification standards to check the instrumental method. These standards may be the same as the LMCS

standards used in routine QC. In addition, a LMCS standard may be processed through the sample preparation (acid digestion) as a means of checking the entire analytical system. For radiochemical methods, bracketing the sample preparation batch with a standard does not enhance the confidence of the results significantly. It is not a cost effective method for verifying instrument calibrations since it reduces the number of samples that can be included in the preparation batch. Appendix A identifies those methods where either calibration verification or LMCS standards are routinely run before and after a batch. Other methods include at least one standard per analytical or preparation batch. These standards are normally evaluated and reported as a part of the analytical batch summary sheet or other method specific reporting form. The guidance for standards in this TPP and Appendix A are intended to meet the recent ECN requirements and will be used to characterize samples in this program.

The 102-AP states that the "reference samples" results shall be within three standard deviations of the mean result and that the mean shall be within 25% of the known concentration. This is normally achieved. However, the neptunium method is normally biased 20-30% low and may not always meet the latter criteria. Because the neptunium concentrations are normally very low (below detection limits) it may not be reasonable to rerun standards and samples when this criteria is not achieved. This condition may also exists for I-129 results.

Not all standards can be made from National Institute & Standard Technology (NIST) traceable materials because NIST does not provide all the standard materials necessary. Standards may be prepared using reagent grade chemicals and standardized using acceptable techniques.

4.3 BLANKS

Two types of blanks are normally analyzed with samples. If the method requires a special preparation, such as acid digestion, a preparation blank is run with each batch of samples to evaluate contamination from the preparation process. The preparation batch may be split into two or more analytical batches, depending on the number of samples and the method. In addition to the preparation blank, a method (reagent) blank is run with each analytical batch when appropriate for the method. A preparation blank may not be run with each analytical batch. The method blank may be referred to as a calibration blank for some methods--such as ICP.

4.4 DUPLICATES

The laboratory will take and analyze duplicate aliquots for each sample (100% duplicates) submitted for analysis. The test plans indicate (Section 8.2 for 105-AP and 106-AP and Section 6.2 for 102-AP) that if the relative percent difference (RPD) for the duplicates exceeds three times the measurement error for the method then the results should be checked and the samples reanalyzed if necessary. If the standard deviation for the standard (LMCS) or reference sample results is used for the measurement error, a significant number of reruns may result because (1) these standards do not reflect errors caused by sample heterogeneity or all sample preparation steps and (2) the error of the difference of two results and RPD value is greater

than the error of a single observation. If the concentration of the analyte is near the detection limit the RPD criteria cannot be met. Relative percent difference criteria should only be used to evaluate analytes with concentrations 5 to 10 times the detection limit of the method. The laboratory will rerun duplicate analyses only if the results are greater than 5 to 10 times the detection limits. Only one rerun will be performed to evaluate the results. If the data still does not meet the acceptance criteria and a reasonable analytical cause for the results cannot be identified, no further analyses will be performed until the data can be reviewed by the customer to verify that additional analyses will be needed.

The test plans (Section 7.1 for 105-AP and 106-AP and Section 3.1 and 3.2 of 102-AP) indicate that duplicate aliquots of samples will be submitted for analysis. Unless the program wants (1) the samples analyzed in quadruplicate, (2) to evaluate the subsampling errors for both the hot cell and analytical lab or (3) wants to run a 100% blind sample program, it is recommended that only one subsample be submitted for the required analyses. The laboratory is planning to take two aliquots from each sample submitted for analysis based on recent ECN changes to the plans and comments on this TPP.

Duplicate samples (samples taken from the same location in the tank) are being taken for organics in all three test plans. In addition, duplicate samples are being taken for some inorganic and radiochemical analyses for the 102-AP characterization plan as noted in the summary in Appendix A.

4.5 SPIKES

The test plans (Section 5.1 for 105-AP and 106-AP and Section 10.2 for 102-AP) indicate that either "some spikes will be needed" or that a spike is required for each batch of samples. Spikes are normally used to evaluate if the sample matrix is impacting the performance of the method. One spike for each batch may not provide information on all matrices if the batch contains samples with two types of matrices. On the other hand, spiking each batch may result in an excessive number of spikes if the same sample matrix requires more than one batch or if small batch sizes are processed.

Because the wastes in the GTF test plans are liquids and relatively homogenous (one matrix), one or two spikes per tank may be adequate for the evaluation. If a tank is expected to be layered, spiked samples from each layer may be desirable. Samples from different risers may be considered to be from different matrices. Spiking alternatives include: (1) only the composite, (2) the composite and one individual sample, (3) the composite and a sample from each layer or (4) the composite and a sample from each riser. Based on Grout Technologys' response to the draft TPP, the spikes will be performed only on each composite. The samples to be spiked should be identified on the RSA. Spike concentrations must be at least 25% of the analyte concentration in the sample to achieve accurate measurements. Spikes are primarily applicable only to analytes whose concentrations are <1000 ug/mL. If the analyte concentration is greater than 1000 ug/mL, then the serial dilution results for some methods (ICP/AES) may be evaluated for potential indications of interferences. Spikes may be added during the sample preparation (acid digestion) or after the preparation (post digestion). Only the ICP/AES samples are spiked during digestion. Analytes for which spikes are appropriate are identified in Appendix A.

At least one TCLP extract will be spiked for a given grout formulation when more than one TCLP test is done for that formulation. As discussed earlier in Section 3.3 of this TPP, the TCLP spiking point (step) is being discussed with the EPA. Toxic characteristic leach procedure extracts may be spiked at two points (before acid preservation or before acid digestion) for evaluation of the procedure and to support decisions made with the data.

4.6 DETECTION LIMITS

Detection (quantitation) limit requirements are primarily addressed only in Section 4 of the QAPjP for the 102-AP test plan. Based on the performance of the laboratory methods on simple matrices using normal sample sizes, the quantitation limits specified for the inorganic and radiochemical methods should be achievable. However, the ability to achieve the desired detection limits can be impacted by sample matrices and limited sample sizes.

Many of the laboratory detection limits are based on the lowest calibration point or the limitation of some other measurement parameter. In most cases this approach results in a conservative estimate of the detection limits, assuming no matrix effects. Detection limits for ICP are determined using the more rigorous CLP procedure in which standards are measured repeatedly near the detection limit and the detection limit is based on the standard deviation of these measurements. This approach still does not account for problems that may be caused by the matrix. Radiochemical detection limits are dependent on the background of the detector and the count times used. These backgrounds are reported for each analysis and may be used to evaluate the detection limit for the detector using standard radiochemical counting practices.

More rigorous detection limits may be needed when it is necessary to prove that the results are below a specified level. Several methods exists for determining detection limits, all of which have a degree of uncertainty. If the sample matrix is expected to impact the detection limits, the detection limits for a method should be determined on the matrix (without the contaminate of interest) by performing multiple standard additions to the matrix near the estimated detection limit. This type of detection limit determination will require an increase in resources and increase the cost of analyses; therefore, the need for this type of detection limit should be evaluated carefully. More rigorous detection limit evaluations will not be performed unless the data indicate that the measurement limit for the method is inadequate or questionable or the program feels that more data is needed to substantiate the results.

4.7 HOLDING TIMES

Holding time requirements and limitations are not addressed in the GTF test plans. Only the 102-AP plan, Section 9.2 states that holding times will be validated by HASM. Holding times are address in the grout SOW.

Regulatory holding times for the ICP/AES metals are achievable. However, the 28 day holding time for Hg on composites may be difficult to achieve depending on the time required in starting the program, sample receipt, and composite preparation. Holding times for anions are generally

not achievable, however, the holding time sensitive anions are normally present at such high concentrations that failure to meet the holding times is not expected to significantly impact the results.

SW-846 holding times are designed for water samples in which trace components can be lost by various mechanisms: evaporation, biological, chemical, and physical interactions. The highly radioactive nature of Hanford wastes makes the use of standard preservation techniques impossible. In addition, the complexity of sampling, transporting, and storing of wastes makes some of the shorter holding times impossible to achieve. Because the anions (nitrate and nitrite) which have short holding times are normally present in such high concentrations, any losses caused by longer holding times are not as significant as if they were trace components in water.

The laboratory recommends that the program may want to provide better holding time instructions in two areas: (1) mercury analysis and (2) analysis of ANSI leachates. The holding time requirement for Hg in the TCLP procedure requires that the TCLP extraction be performed within 28 days from field collection. This time cannot be achieved because of the 28 day minimum curing time for the grout specimens, unless the "field collection" is considered to start at the completion of the grout curing. Because of potential losses, Hg TCLP results below the TCLP limits could be questioned. Therefore, it may be important to ensure that the Hg analysis performed on the GTF feed waste (102-AP) be done within the 28 day holding time. The samples for Hg will be submitted as rapidly as possible by the PSC and the holding time requirement clearly identified on the Request for Special Analysis (RSA) form. The laboratory will attempt to achieve the Hg holding times for the 102-AP samples.

Leachates from the ANSI test will contain anions (nitrate and nitrite) at lower concentrations which could significantly change with time. It is recommended that the program consider requesting that these analyses be completed in a specified time.

5.0 REPORTS AND DELIVERABLES

5.1 STATUS REPORTS

Each of the three test plans require monthly status reports to Grout Technology. The monthly status report will detail the progress to date and the projected completion date for the various activities. This shall be the responsibilities of the laboratory project coordinator.

5.2 SPECIAL REPORTS

In addition to the monthly status reports, special reports shall be prepared within a week of observing the following conditions in the testing:

- Listed organic constituents exceeding concentration limits in the plans.
- Grout specimen compressive strength results <500 psi.
- TCLP metal concentrations in waste that exceed 20 times the TCLP extraction limits.
- Leach indices for nitrate and nitrite at less than 6.

If these limits are exceeded, the condition shall be reported by phone and followed by an interim report to the responsible Grout Technology engineer.

5.3 ANALYTICAL DATA REPORTS

The analytical data reports for the candidate tanks (105-AP and 106-AP) will be different than those for the grout feed tank (102-AP). With the exception of the organic analyses, which should include all calibrations and raw data, the candidate tank data reports will not require a data package . The 102-AP data report (Section 9 of the QAPjP) indicates that a full data package with calibrations and raw data to support all analyses <u>may</u> be required. If the program wants the data package to be reviewed by QA groups and validated by HASM, all the raw data will be required. Based on Grout Technology review of the draft TPP, a full data package with all supporting raw data will be prepared for the 102-AP.

Two of the test plans (Section 5.4 for 105-AP and 106-AP) indicate that the data should be reported on CLP type forms. All three of the test plans recommend a CLP form (Section 15 for 105-AP and 106-AP and Section 9.3 for 102-AP) for reporting results. Only the ICP and organic data are reported on multiple page forms similar to those used in the CLP program. The form identified in the plan is inadequate for reporting all the data (results and QC information) for each test. If this data must be evaluated by the Process Laboratory and Technology statistics team and Grout Technology within 30 to 60 days, the data will have to be reported in preliminary reports before full validation of the data package.

Preliminary report data for the program will be provided through the use of LCCS reports or copies of the batch summary reports. The program must recognize that the use of these preliminary reports carries a risk because the data has not gone through the entire laboratory review process and errors may lead to data changes at a later date. The Process Laboratory and Technology statistics team, PSC, and Grout Technology will use these preliminary data to determine if additional analyses and formulation and product testing is required. The analytical project coordinator will prepare a narrative and summary for the final 105-AP and 106-AP results. A full data package for tank 102-AP, including a summary report, will be prepared after completion of all the tests. Analytical results for the 90 day ANSI leach tests will not be included in the initial data package. The length of time to complete the tests makes it impossible to meet schedule requirements. Evaluation of the ANSI leach tests, grout physical testing, and statistical evaluations of the characterization data may be documented in different reports than the analytical data packages and summaries.

Data requirements for the original 106-AP plan ANSI leach tests identifies 13 analytes for determination including sodium (Section 7.12.2). The ANSI tests for 102-AP include only nitrate, nitrite and pH. The ANSI leach test requirements have been modified by ECN to be nitrate, nitrite, and pH for all tanks. Technetium-99 and Am-241 will be included if the concentration of these isotopes significantly exceed the detection limits in the wastes used to make the grout specimen.

5.4 BUDGET AND SCHEDULE ESTIMATES

The schedules provided in this section are based on the times required to meet the Hanford Federal Facility Agreement and Consent Order. The grout program schedule needs may differ from this requirement and will be negotiated on detailed schedules. Priorities for the three tanks may require that characterization of the 102-AP precede that of the 105-AP. More detailed schedules will be prepared by Laboratory Schedule and Work Control as better defined starting dates and priorities are established.

The schedules and length of time to complete the characterization may exceed the dates and times shown. Completion of the characterization is contingent on available laboratory analytical capacity and TWRS priorities.

Estimated Grout Schedule

Tank	Start Date	Completion Date	Cycle Days
105-AP	3/12/93	7/29/93	140
102-AP	4/20/93	9/7/93	140
106-AP	5/5/93	9/21/93	140

The statement of work for the program requires data packages for the different analytical operations within 85 days of sample receipt and 45 days for the treatment tests. Analytical and testing reports are required 30 to 45 days after completion of analyses. The treatment tests require a 28 day

curing time for the grout specimens in addition to a 5 day ANSI leach time. This will make meeting the 45 day requirement difficult without the highest priority and full support. The 85 days for data packages is also considerably shorter than has been achieved for the Single Shell Tank Characterization Program. The ability to achieve this schedule is questionable.

Cost requirements for characterization of grout waste feed for Fiscal Year 1993 are being negotiated with the customer.

Therefore, the laboratory cannot commit to time periods less than those required by the Hanford Federal Facility Agreement and Consent Order. The exact schedule and contents of the deliverables will be negotiated with the customer.

6.0 REFERENCES

40 FR 4440, 1990, "Hazardous Waste Management System, Testing and Monitoring Activities," *Federal Register*, Vol. 55, No. 27, pp. 4440-4444, (February 8), proposed rules.

DOE-RL, 1992, *Grout Treatment Facility Waste Permit Application*, DOE/RL-88-27, Rev. 2, U.S. Department of Energy Richland Field Office, Richland, Washington.

Hendrickson, D. W., 1991, *Grout Treatment Facility Waste Feed Acceptance Criteria*, WHC-SD-WM-RD-019, Rev. 1, Westinghouse Hanford Company, Richland, Washington.

APPENDIX A

GROUT ANALYTICAL REQUIREMENTS

Tank 105-AP Grout Analysis Requirements.

Test/Analyte	STD	Individual			Composite			ANSI-16.1			Drain Liq.			Field Blank			Individ. Part.		
		SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK
								Not Applicable			Not Applicable						Not Applicable		
SPL PREP																			
Acid Digest	B&A	A	A		A	A	A												
METALS																			
ICP	B&A	A	A		A	A	A												
As	B&A				A	A	A												
Se	B&A	A	A		A	A	A												
Hg	B&A				A	A	A												
U	1/Batch				A	A	A												
ANIONS/WET																			
IC	B&A	D(w)	D(w)		D(w)	D(w)	P												
NH4	1/Batch				D(A)	D(A)	P												
Wt% H2O	1/Batch	D	D		D	D													
OH	1/Batch				D	D													
pH	1/Batch																		
CO3	1/Batch	D(w)	D(w)	P															
TOC	1/Batch	D(w)	D(w)		D(w)	D(w)	P												
CN	1/Batch				D	D	D												
PHYSICAL																			
Density	1/Batch	D	D		D	D													
RAD CHEM																			
GEA	1/Batch	D(A)	D(A)		D(A)	D(A)													
Cs-137		D(A)	D(A)		D(A)	D(A)													
Cs-134		D(A)	D(A)		D(A)	D(A)													
Co-60		D(A)	D(A)		D(A)	D(A)													
CePr-144		D(A)	D(A)		D(A)	D(A)													
Sb-125		D(A)	D(A)		D(A)	D(A)													
RuRh-106		D(A)	D(A)		D(A)	D(A)													
Nb-94		D(A)	D(A)		D(A)	D(A)													
Isotopes>DL		D(A)	D(A)		D(A)	D(A)													
Alpha																			
Pu-239/240	1/Batch	D(A)	D(A)	T															
Pu-238		D(A)	D(A)	T															
Pu-241					To be calculated														
Am-241	1/Batch	D(A)	D(A)	T	D(A)	D(A)	T												
Cm-244		D(A)	D(A)	T	D(A)	D(A)	T												
Np-237	1/Batch	D(A)	D(A)	D(A)	D(A)	D(A)	D(A)												
Beta																			
Sr-90	1/Batch	D	D	C	D	D	C												
Tc-99 (LSC)	1/Batch	D	D	D	D	D	D												
I-129 (LEPS)	1/Batch	D	D	C	D	D	C												
H-3 (LSC)	1/Batch				D(w)	D(w)	D(w)												
C-14 (LSC)	1/Batch	D(w)	D(w)	D(w)															
Grout Test																			
TCLP					Not requested														
ANSI					Not requested														
Viscosity					Not requested														
Crit. Flow					Not requested														
Frict. P Drop					Not requested														
Density					Not requested														
Gel Strength					Not requested														
Drain Liquid					Not requested														
Comp. Streng.					Not requested														
Organics																			
VOA		PNL on duplicate samples												PNL					
Semi-VOA		PNL on duplicate samples																	
Complexants		PNL on duplicate samples																	

Tank 106-AP Grout Analysis Requirements

Test/Analyte	STD	Individual			Composite			ANSI-16.1			Drain Liq.			Field Blank			Individ. Part.		
		SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK
																	Not Applicable		
SPL PREP																			
Acid Digest	B&A	A	A		A	A	A												
METALS																			
ICP	B&A	A	A		A	A	A				A	A							
As	B&A				A	A	A												
Se	B&A	A	A		A	A	A												
Hg	B&A				A	A	A												
U	1/Batch				A	A	A												
ANIONS/WET																			
IC	B&A	D(w)	D(w)		D(w)	D(w)	P	D(w)	D(w)		D(w)	D(w)							
NH4	1/Batch				D(A)	D(A)	P												
Wt% H2O	1/Batch	D	D		D	D													
CH	1/Batch				D	D													
pH	1/Batch							D											
CO3	1/Batch	D(w)	D(w)	P															
TOC	1/Batch	D(w)	D(w)		D(w)	D(w)	P												
CN	1/Batch				D	D	D												
PHYSICAL																			
Density	1/Batch	D	D		D	D													
RAD CHEM																			
GEA	1/Batch	D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Cs-137		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Cs-134		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Co-60		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
CePr-144		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Sb-125		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
RuRh-106		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Nb-94		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Isotopes>DL		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Alpha																			
Pu-239/240	1/Batch	D(A)	D(A)	T	D(A)	D(A)	T												
Pu-238		D(A)	D(A)	T	D(A)	D(A)													
Pu-241		To be calculated																	
Am-241	1/Batch	D(A)	D(A)	T	D(A)	D(A)	T	D(A)	D(A)	T									
Cm-244		D(A)	D(A)	T				(If >DL)											
Np-237	1/Batch	D(A)	D(A)	D(A)															
Beta																			
Sr-90	1/Batch	D	D	C	D	D	C												
Tc-99 (LSC)	1/Batch	D	D	D	D	D	D	D	D	D									
I-129 (LEPS)	1/Batch	D	D	C	D	D	C	(If >DL)											
H-3 (LSC)	1/Batch				D(w)	D(w)	D(w)												
C-14 (LSC)	1/Batch	D(w)	D(w)	D(w)															
Grout Test																			
TCLP(Metals)					G(A)	G(A)	G(A)												
ANSI					G														
Viscosity					X														
Crit. Flow					X														
Frict. P Drop					X														
Density					X														
Gel Strength					X														
Drain Liquid					X														
Comp. Streng.					G														
Organics																			
VOA		PNL on duplicate samples												PNL					
Semi-VOA		PNL on duplicate samples																	
Complexants		PNL on duplicate samples																	

Tank 102-AP Grout Analysis Requirements

Test/Analyte	STD	Individual			Composite			ANSI-16.1			Drain Liq.			Field Blank			Individ. Part.		
		SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK	SPL	DUP	SPK
SPL PREP																			
Acid Digest	B&A	A	A		A	A	A												
METALS																			
ICP	B&A	A	A		A	A	A	Dropped			A	A					A	A	
As	B&A				A	A	A										(Na Only on ICP)		
Se	B&A	A	A		A	A	A				A	A							
Hg	B&A				A	A	A												
U	1/Batch				A	A	A												
ANIONS/WET																			
IC	B&A	D(w)	D(w)	P	D(w)	D(w)	P	D(w)	D(w)		D(w)	D(w)					D(w)	D(w)	
NH4	1/Batch				D(A)	D(A)	P				D(A)	D(A)					(PO4 only on IC)		
Wt% H2O	1/Batch	D	D		D	D													
CH	1/Batch	D	D		D	D					D	D							
pH	1/Batch							D											
CO3	1/Batch	D(w)	D(w)	P															
TOC	1/Batch	D(w)	D(w)		D(w)	D(w)	P				D(w)	D(w)							
CN	1/Batch				D	D	D												
PHYSICAL																			
Density	1/Batch	D	D		D	D													
RAD CHEM																			
GEA	1/Batch	D(A)	D(A)		D(A)	D(A)					D(A)	D(A)					D(A)	D(A)	
Cs-137		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)					D(A)	D(A)	
Cs-134		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Co-60		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
CePr-144		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Sb-125		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
RuRh-106		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Nb-94		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Isotopes>DL		D(A)	D(A)		D(A)	D(A)					D(A)	D(A)							
Alpha																			
Pu-239/240	1/Batch	D(A)	D(A)	T															
Pu-238		D(A)	D(A)	T															
Pu-241		To be Calculated																	
Am-241	1/Batch	D(A)	D(A)	T	D(A)	D(A)	T	D(A)	D(A)	T									
Cm-244		D(A)	D(A)	T				(If >DL)											
Np-237	1/Batch	D(A)	D(A)	D(A)															
Beta																			
Sr-90	1/Batch	D	D	C	D	D	C				D	D	C						
Tc-99 (LSC)	1/Batch	D	D	D	D	D	D	D	D	D	D	D	D						
I-129 (LEPS)	1/Batch				D	D	C	(If >DL)			D	D	C						
H-3 (LSC)	1/Batch				D(w)	D(w)	D(w)												
C-14 (LSC)	1/Batch	D(w)	D(w)	D(w)															
Grout Test																			
TCLP(Metals)					G(A)	G(A)	G(A)												
ANSI					G														
Viscosity					X														
Crit. Flow					X														
Frict. P Drop					X														
Density					X														
Gel Strength					X														
Drain Liquid					X														
Comp. Streng.					G														
Organics																			
VOA		PNL on duplicate samples												PNL					
Semi-VOA		PNL on duplicate samples																	
Complexants		PNL on duplicate samples																	

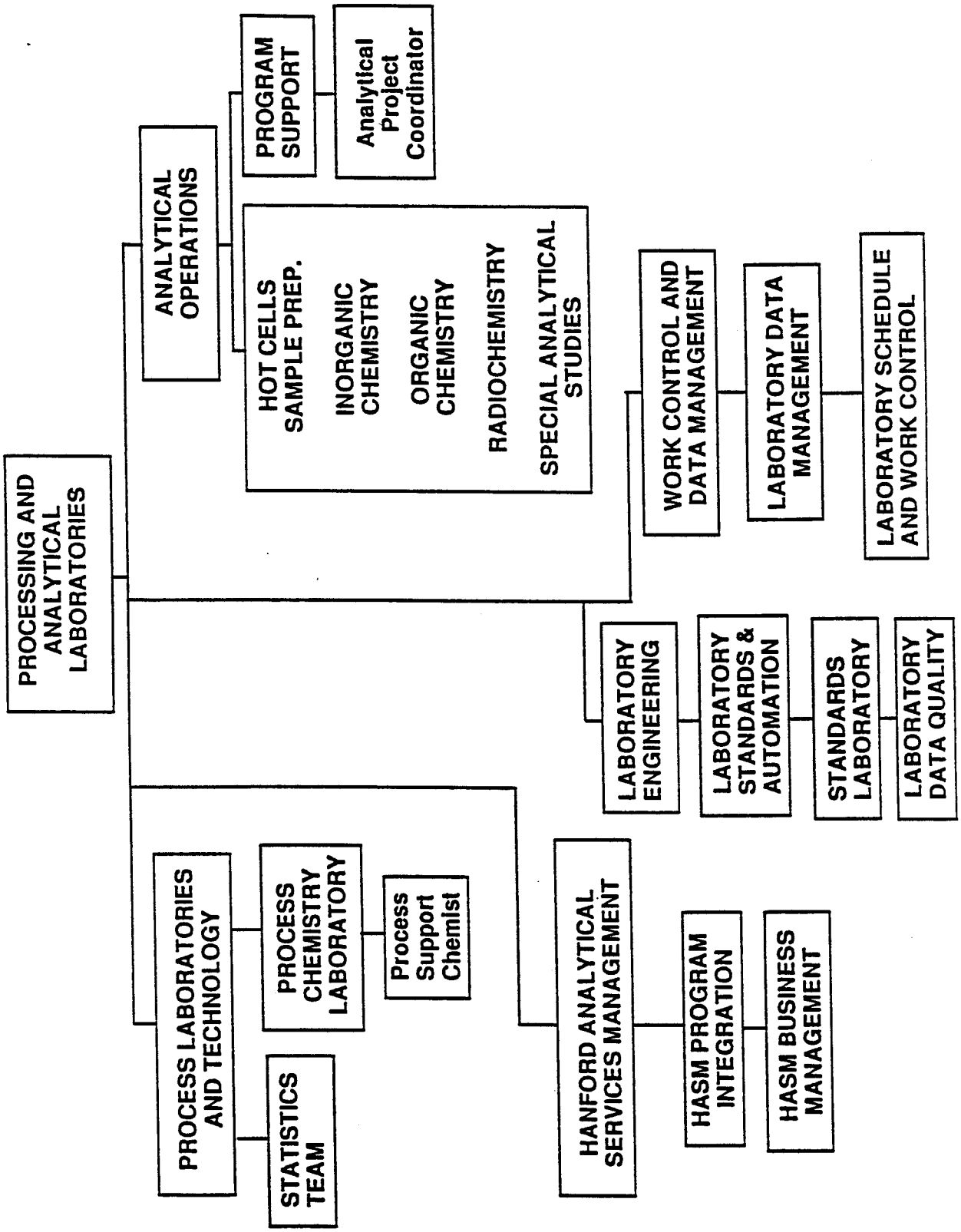
Grout Analytical Requirements Technical Information and Abbreviations

		Definitions and Technical Information				
		105- AP	**106-AP**	**102-AP**		
Individual samples		12	6	9Full, 6Part		
Risers		3	3	3		
Depths		4	2	5		
Composites		1	1	1		
Drainable Liq. Composites			2	2	(If material available)	
Spikes		1/Riser or Alt.	1/Riser or Alt.	1/Riser or Alt.		
Organic samples to PNL		6	6	6+Dup		
Organic field/hotcell blank		1	1	2		
Dup. Samples for Inorg. & Rad				3		
Dup. Analyses for Inorg. & Rad		100%	100%	100%		
Grout Tests		No	Yes	Yes		
			6-prepared	12- prepared		
			3- Comp Str.	5- Comp Str.		
			2-TCLP+BLK	5-TCLP+BLK		
			1-ANSI +BLK	3-ANSI +BLK		
				4-Gas Generation + 4 Blanks		
ICP	**Analytes**	**105-AP**	**106-AP**	**102-AP**	**ANSI**	**Gas**
	Ag*	x	x	x	**ANALYTES**	**Generation**
	Al	x	x	x		**ANALYTES**
	Ba*	x	x	x	Nitrate	
	Be	x	x	x	Nitrite	N20
	Cd*	x	x	x	pH	H2
	Cr*	x	x	x	If composite>	CH4
	Fe	x	x	x	detection limit	N2
	Na	x	x	x	then	O2
	Ni	x	x	x	Am-241	
	P	x	x	x	TC-99	
	Pb*	x	x	x		
	Sb	x	x	x		
	* TCLP Metals + As, Se, and Hg by AA					
Abbreviations						
A - Analysis is performed on acid digestion of sample						
B & A - Standards are run before (B) and after (A) a batch of samples						
D(A) - Analysis performed on direct sample or a sample that has been preserved or diluted in acid						
D(W) - Analysis performed on direct sample or a sample that has been diluted in water						
P - Spike is added to diluted or already prepared sample						
T - Method uses radioactive tracer in place of spike to estimate method recovery						
C- Method uses nonradiactive standard in place of spike to estimate method recovery						
DL - Detection limit						
LSC - Liquid scintillation counting						
LEPS - Low energy photon spectroscopy						
G - Test run on grouted waste or cured grout						
G(A) - Test run on grout specimen and acid digest done on extract						
PART. - Individual samples for which only a PARTial list of analytes determined						
Dup. - Duplicate sample or analysis						
Alt. - Alternate samples for spiking, e. g. layers, composites etc.						
Note: Only 1 spiked individual sample is required for CO3 analysis						

APPENDIX B

LABORATORY ORGANIZATION AND KEY PERSONNEL

Laboratory Organization



222-S Analytical Laboratory Key Personnel List

INDIVIDUAL	ORGANIZATION	RESPONSIBILITY
ML Bell	PAL	Manager of Processing and Analytical Laboratories
DA Dodd	PAL	Technical Director
AP Hammitt	Process Chemistry Lab	Process Support Chemist (Single Point of Contact)
MJ Duchsherer	Program Support	Analytical Project Coordinator
WI Winters	Inorganic Chemistry	Acting Manager of Inorganic Chemistry Group
BR Wels	Inorganic Chemistry	ICP/OES Chemist
JS Lachut	Inorganic Chemistry	TOC, Cyanide and Wet Chemical Methods Chemist
SD Bolling	Inorganic Chemistry	Ion Chromatography
RK Fuller	Inorganic Chemistry	Sample Preparations and Analytical Hot Cell Chemist
RE Skoda	Inorganic Chemistry	CVAA, HYAA and GFAA Chemist
RR Grabbe	Organic Chemistry	Manager of Organic Chemistry Group
SG Metcalf	Organic Chemistry	Volatile Organic Chemist
DR Hansen	Organic Chemistry	Semi-Volatile Organic Chemist
EF Riedel	Radiochemistry	Manager of Radiochemistry Group and Counting Room
SA Catlow	Radiochemistry	Radiochemical Separations Chemist
RA Jones	Radiochemistry	Alpha/Beta Counting Room Chemist
AK Dasgupta	Radiochemistry	Gamma Energy Counting Room Chemist
LL Lockrem	Special Studies	Manager of Special Studies
RP Marshall	Analytical Operations	Manager of Analytical Operations
JG Kristofzski	Program Support	Manager Program Support
JR Prilucik	Analytical Operations	Manager 222-S Hot Cells and Sample Preparation
Shift Manager	Analytical Operations	Off-hour point of contact

222-S Analytical Laboratory Key Personnel List (continued)

SL Cobb	Hot Cell and Sample Prep.	Sample Custodian
JP Sloughter	Process Lab and Technology	Manager of Process Laboratories and Technology
TL Welsh	Process Lab and Technology	Statistics Team
JR Jewett	Process Chemistry Lab	Manager of Process Chemistry Laboratory
DB Bechtold	Process Chemistry Lab	Particle Size, Adiabatic Calorimetry, Thermal Analysis
WD Leggett	Laboratory Engineering	Manager of Laboratory Engineering
TG Ibsen	Lab Standards and Automation	Manager Laboratory Standards and Automation - LIMS systems
WW Baird	Standards Laboratory	Manager of Standards Laboratory
DR Jordan	Work Control and Data Management	Manager of Work Control and Data Management
PK Vacca	Lab Data Management	Manager Laboratory Data Management
L R Webb	Lab Data Management	Records Specialist for DST/SST Data Packages
JL Zwicker	Lab Scheduling & Work Control	Manager Lab Scheduling and Work Control
JG Rukavina	Lab Scheduling & Work Control	Activity Admistrator - Prepares Schedules and Status of Work
CR Stroup	HASM	Acting Manager of HASM
SM Joyce	HASM Program Integration	Manager of Laboratory Programs- long term schedules and budgets
RA Spohr	HASM Business Management	Manager HASM Business Management
LH Taylor	Laboratory/Data Quality	Manager of Laboratory and Data Quality
LP Markel	Laboratory/Data Quality	Quality Assurance Officer - QA/QC Review

Grout Analytical Requirements Technical Information and Abbreviations

		Definitions and Technical Information				
		105- AP	**106-AP**	**102-AP**		
Individual samples		12	6	9Full, 6Part		
Risers		3	3	3		
Depths		4	2	5		
Composites		1	1	1		
Drainable Liq. Composites			2	2	(If material available)	
Spikes		1/Riser or Alt.	1/Riser or Alt.	1/Riser or Alt.		
Organic samples to PNL		6	6	6+Dup		
Organic field/hotcell blank		1	1	2		
Dup. Samples for Inorg. & Rad				3		
Dup. Analyses for Inorg. & Rad		100%	100%	100%		
Grout Tests		No	Yes	Yes		
			6-prepared	12- prepared		
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			1-ANSI +BLK	3-ANSI +BLK		
				4-Gas Generation + 4 Blanks		
ICP	**Analytes**	**105-AP**	**106-AP**	**102-AP**	**ANSI**	**Gas**
	Ag*	x	x	x	**ANALYTES**	**Generation**
	Al	x	x	x		**ANALYTES**
	Ba*	x	x	x	Nitrate	
	Be	x	x	x	Nitrite	N20
	Cd*	x	x	x	pH	H2
	Cr*	x	x	x	If composite>	CH4
	Fe	x	x	x	detection limit	N2
	Na	x	x	x	then	O2
	Ni	x	x	x	Am-241	
	P	x	x	x	TC-99	
	Pb*	x	x	x		
	Sb	x	x	x		
	* TCLP Metals + As, Se, and Hg by AA					
Abbreviations						
A - Analysis is performed on acid digestion of sample						
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DL - Detection limit						
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LEPS - Low energy photon spectroscopy						
G - Test run on grouted waste or cured grout						
G(A) - Test run on grout specimen and acid digest done on extract						
PART. - Individual samples for which only a PARTial list of analytes determined						
Dup. - Duplicate sample or analysis						
Alt. - Alternate samples for spiking, e. g. layers, composites etc.						
Note: Only 1 spiked individual sample is required for CO3 analysis						

APPENDIX C

222-S LABORATORY PROCEDURES

222-S Laboratory Procedures in Support of Grout Treatment Facility

Test Parameter	Procedure Number	Rev No.	Title
PREPARATION			
Acid Digestion Soli	LA-505-159	B-0	Acid Digest. of Sediments, Sludges, and Soils for ICP and AA Analysis
Water Digestion	LA-504-101	B-0	TaNk Core Water Soluble Solids
Acid Digest Liquids	LA-505-158	A-2	Acid Digest of Aqueous Samples and Extracts
			for Total Metals Analysis By FLAA or ICP Spectroscopy
METALS			
ICP - metals	LA-505-151	C-0	ICP Emission Spectrometer Method for Trace Element Analysis
CVAA- Hg	LA-325-104	A-0	Mercury Analysis by Atomic Absorption (Manual Cold Vapor Technique)
ANIONS/OTHERS			
Total Cyanide	LA-695-102	B-O	Microdistillation and Spectrophotometric Determination of CN
ANIONS -IC	LA-533-105	B-1	Anion Analysis on Dionex Model 4000i
NO2- SPEC	LA-645-001	A-4	Spectrophotometric Determination of Nitrite
pH	LA-212-101	C-4	Set Up and Standardization of pH Meter and Glass Electrode
pH	LA-212-102		Determination of pH Direct Measurement
pH	LA-212-103	B-1	Determination of pH on SST Core Samples
OH	LA-661-102	F-1	Determination of OH Ion in Solutions by Potentiometric Titration
NH3	LA-634-102	D-0	Ammonia by Kjeldahl
TOC water	LA-344-105	B-2	Determination of Carbon in Solutions by Combustion and Coulometry
TIC (CO3)	LA-622-102	B-2	Determination of Carbonate in Solutions by Coulometry
Radiochemical			
Uranium	LA-925-106	B-0	Determination of Uranium by Laser Fluorimetry
GEA	LA-508-052	B-4	Gamma Energy Analysis on the Canberra Jupiter System
	LA-548-121	D-0	Preparation of Sample Mounts for GE(Li) GEA - Low Level
Total Alpha	LA-508-101	D-0	Low Level Alpha and Beta in Water Samples
	LA-508-104	A-3	Total Alpha Counting by Alpha Proprotional Counters
Total Beta	LA-508-101	D-0	Low Level Alpha and Beta in Water Samples
	LA-508-114	A-1	Operation of Gamma Products Alpha/Beta Counting System
Pu-238,239/240	LA-503-156	D-0	Determination of Pu and Am by Ion Exchange and Solvent Extraction
	LA-508-051	A-3	Alpha Energy Analysis using the Canberra Jupiter Systems
Am/Cm	LA-503-156	D-0	Determination of Pu and Am by Ion Exchange and Solvent Extraction
Np-237	LA-933-141		Determination of Np-237 by TiOA/TTA Extraction and Alpha Counting
Sr-90	LA-220-101	D-0	High Level Strontium-89,90 in Aqueous Samples
Tc-99	LA-438-101	D-1	Determination of Tc-99 by Solvent Extraction and Liq. Scint Counting
I-129	LA-378-103	B-1	Determination of Iodine-129 in Waste Tank Samples
C-14	LA-348-104	B-0	C-14 in Small Volume Samples by Persulfate Oxidation and Liq. Scint.
H-3	LA-218-114	A-1	Tritium by Lachat Micro-Dist. and Liquid Scintillation Counting (LS)
	LA-508-121	B-0	Operation of the Beckman LS-5801 Liquid Scintillation System
Se-79	LA-365-132		Determination of Se-79
Physical Methods			
Wt% Water	LA-564-101	E-1	Percent Water in Tank Farm Samples and Pilot Evaporator Spls.
Density (liquids)	LA-510-112	C-2	Specific Gravity of High Beta Gamma Caustic Samples
Organic Methods			
Semi Volatile	PNL		
Complexants	PNL		

222-S Laboratory Procedures in Support of Grout Treatment Facility (continued)

Test Parameter	Procedure Number	Rev No.	Title
Grout Tests			
Dry Blend Prep	LA-519-170	A-2	Preparation of Homogeneous Dry Solids Blend for a Grout Formulat
Grout Mix Prep	LA-519-171	A-2	Grout Mixtures Preparations
Moisture	LA-519-172	A-2	Moisture in Non-Liquid Grout Admixtures
	LA-519-173	A-2	Density by Mud Balance
Viscosity	LA-519-174	A-1	Determination of Viscosity Using the Fann Viscometer
Critical Flow Rate	LA-519-174	A-1	Determination of Viscosity Using the Fann Viscometer
Frict. Press. Drop	LA-519-174	A-1	Determination of Viscosity Using the Fann Viscometer
10 Min Gel Strength	LA-519-175	B-0	Grout Gel Strength-Fann Viscometer
Bleed Water	LA-519-176	B-0	Density and Bleed Water of Grout
Setting Time	LA-519-177	A-1	Setting Time of Grouts - ACME Penetrometer
Setting Time	LA-519-178	A-1	Setting Time of Grouts - Vicat Needle
Compressive Strength	LA-519-179	A-0	Compressive Strength of Cement- Based Grouts
Viscosity	LA-519-180	A-0	Determination of Viscosity Using the Bohlin Viscometer
Pulse Velocity	LA-519-181	A-0	Determination of Pulse Velocity Through Cured Grout
TCLP	LA-544-131	A-0	Toxic Characteristics Leaching Procedure (TCLP)- Hot Cell
TCLP	LA-544-132	A-0	Toxic Characteristics Leaching Procedure (TCLP)- Non Radioactive Sampl
TCLP	LA-544-133	A-0	Toxic Characteristics Leaching Procedure (TCLP)- Low Activity Samples
ANSI Leach	ANSI 16.1		
Compress Strength	ASTM C-101-86		
Pulse Velocity	ASTM C-591-83		
Lab Operations			
IC Containers	LO-080-114	B-0	Cleaning of Ion Chromatograph and Plastic Containers
ICP Vials	LO-080-115	B-0	Washing Sample Vials for the ICP
Onsite Shipment	LO-090-004	F-0	Packaging Radioactive Material for Onsite Shipment and Recieving
Sample Custodian	LO-090-101	H-0	Sample Receiving and Custodianship - 222-S Laboratory
Labeling	LO-120-001	C-3	Labeling of Standards and Reagents
Balances	LO-140-004	A-2	Routine Operation of Mettler AE Balances
LMCS	LO-150-001	A-0	Laboratory Measurement Control using the LMCS System
OSCR	LO-150-002	B-0	Responses to Off-Standard Condition Reports and Precision Reports
EDP Codes	LO-150-028	B-1	Documenting LMCS EDP Code Parameter Changes
Analytical Cards	LO-150-043	B-2	Analytical Sample Cards - LCCS
Counting Room	LO-150-112	D-0	Administrative Controls for the 222-S Counting Room Operation
Standard Counting	LO-150-115	A-3	Instrument Standards Counting Frequency - 222-S Counting Room
Data Reporting	LO-150-127	A-3	Mathematic Aspects of Checking and Reporting Results
Procedures	LO-150-144	A-3	Issuing and Updating Procedures at the 222-S Analytical Laboratory
Lab Computer			
FORTRAN	LC-400-001	A-2	FORTRAN Coding and Documentation Guidelines
Calculator	LC-400-002	A-2	Programmable Calculator Documentation and Coding Guidelines
BASIC	LC-400-003	A-0	BASIC Coding and Documentation Guidelines
Spreadsheet	LC-400-006	A-1	Spreadsheet Documentation and Guidelines
Control	LC-400-005	B-0	Laboratory Computer Control
Sensitive	LC-400-004	A-0	Control of Sensitive Computer Systems and Applications

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Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	EDT/ECN Only
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