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C. Willingham

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7. Abstract

This Sample Analysis Plan (SAP) covers the sampling of the liquid and gas in fuel canisters at 105 KW. The data obtained will aid in characterizing the contents of the fuel canisters and selecting canister for hot cell evaluations.N/A

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**SAMPLING AND ANALYSIS PLAN
FOR CANISTER LIQUID AND GAS SAMPLING
AT 105-KW FUEL STORAGE BASIN**

February 28, 1995

Prepared by

R. A. Harris
M. A. Green
B. J. Makenas
D. J. Trimble

Westinghouse Hanford Company
P.O. Box 1970
Richland, WA 99352

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ACRONYMS AND ABBREVIATIONS

AEA	Alpha Energy Analysis
COC	Chain-Of-Custody
DI	Desk Instruction
DQOs	Data Quality Objectives
EPA	Environmental Protection Agency
GEA	Gamma Energy Analysis
HPT	Health Physics Technician
IXCs	Ion Exchange Columns
IXMs	Ion Exchange Modules
KBO	K Basin Operations
MDL	Minimum Detection Limit
MWP	Master Work Plan
NFE	Nuclear Fuels Evaluations
OAWH	Operations Analysis and Waste Handling
PNL	Pacific Northwest Laboratories
PQL	Practical Quantification Limit
PS	Process Systems
QA	Quality Assurance
QC	Quality Control
RWP	Radiation Work Permit
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
WHC	Westinghouse Hanford Company

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) details the sampling and analyses to be performed on fuel canisters transferred to the Weasel Pit of the 105-KW fuel storage basin. The radionuclide content of the liquid and gas in the canisters must be evaluated to support the shipment of fuel elements to the 300 Area in support of the fuel characterization studies (Abrefah, et al. 1994, Trimble 1995). The following sections provide background information and a description of the facility under investigation, discuss the existing site conditions, present the constituents of concern, outline the purpose and scope of the investigation, outline the data quality objectives (DQO), provide analytical detection limit, precision, and accuracy requirements, and address other quality assurance (QA) issues.

This project does not fall under the auspice of the Environmental Protection Agency (EPA). Therefore the requirements of QAMS-005/80, *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans* do not apply. An attempt has been made to meet the intent of QAMS-005/80, however, by including appropriate discussions in this plan. Several items normally included in an EPA sanctioned plan are not listed here because they are addressed in the appropriate facility and laboratory procedures and QA plans. These items are listed below.

- Internal laboratory chain of custody and identification system.
- Calibration requirements and frequencies.
- Preventive maintenance requirements.
- Corrective action program.
- Performance and system audits.
- Data reduction techniques.
- Data assessment methods.
- QA reports to management.

1.1 FACILITY BACKGROUND

The 100-K Area is located on the Hanford Site approximately 25 miles northwest of Richland, Washington. At the east and west end of this area are two identical reactors that were constructed between 1952 and 1954, and were built simultaneously. The easternmost reactor is designated 105-KE, while the westernmost reactor is designated as 105-KW. The major support facilities for these reactors were also identical, although a few ancillary structures are shared. The primary systems in each reactor included a pump house, clear well, filtration plant, and large basin for storage of irradiated fuel. The two reactors began operation in 1955 (WHC 1991).

After terminating operations in 1970 and 1971, decontamination and decommissioning were initiated for the 105-KW and KE reactor systems. Modifications and repairs to the fuel storage basin system were subsequently made to accommodate N Reactor fuel. These changes included making the basin cooling system a closed loop system. In 1975, storage of spent N Reactor fuel began in the 105-KE reactor storage basin. Similarly, in 1981, storage of N Reactor fuel began in the 105-KW basin. Storage of irradiated fuel continues today although shipments of fuel to the basins for storage ceased in 1989 (WHC 1991).

1.1.1 Description Of Fuel Storage Basins

The 105-KE and 105-KW fuel storage basins are both constructed of reinforced concrete. The basin pools are rectangular in shape, with a length of 38.1 m (125 ft), width of 20.4 m (67 ft), and depth of 6.4 m (21 ft). The pools are divided into three sections (West Bay, Center Bay, and East Bay), which are separated by concrete walls. The ends of these walls are open to allow communication between the three sections, as shown in Figure 1-1. Water is maintained in each basin at a depth ranging from 4.7 m (15 ft 6 in) to 5.1 m (16 ft 6 in). The bottom of the pool sets approximately 6.1 m (20 ft) below grade.

On the south side of each basin is the Discharge Pickup Chute, where spent fuel rods from each reactor were discharged into the basin for storage. On the east side of East Bay is the Weasel Pit where dose rate assessments were performed on spent fuel rods. On the north side of each basin are three floor drains that have been sealed with concrete. There are two water filtration systems in place in each reactor.

1.1.2 Water Processing System Description

The 105-KE and 105-KW basin water processing systems include temperature control, pH monitoring, sand filters, cartridge filters, ion exchange columns, and recirculation pumps. The pH of the water is maintained between 5.2 and 9.5, in order to control the corrosion of the metals in the basins. The temperature in the basins is maintained near 10°C (50°F) with a maximum allowable operating water temperature of 32.2°C (90°F). This helps to minimize the transfer of radionuclides from the fuel to the water. The pH and temperature control systems together minimize the release of radioactivity contained in the irradiated fuel and the sediment from entering the basin water.

The remainder of the water processing mechanism includes two separate systems (see Figure 1-1). The first system is composed of a recirculating pump, cartridge filter, air-cooled chiller, and ion exchange columns. This system draws the basin water through an 2.4 m (8 ft) deep underwater header pipe located in each bay. The water then passes through a cartridge filter designed to remove particles 5 microns or greater. Water is then routed through a chiller to remove residual decay heat. The chilled water is then passed through ion exchange columns and allowed to return to the fuel basin. The cartridge filters are replaced, as are the ion exchange columns (IXC), on a regular basis to remove the accumulated sediment.

A second system pumps water from near the surface of the water at a rate of 25.2 L/s (400 gpm) through a large sand filter containing approximately 3,900 Kg (8,700 pounds) of sand. The water is then routed through ion exchange modules (IXM), then discharged to the basin.

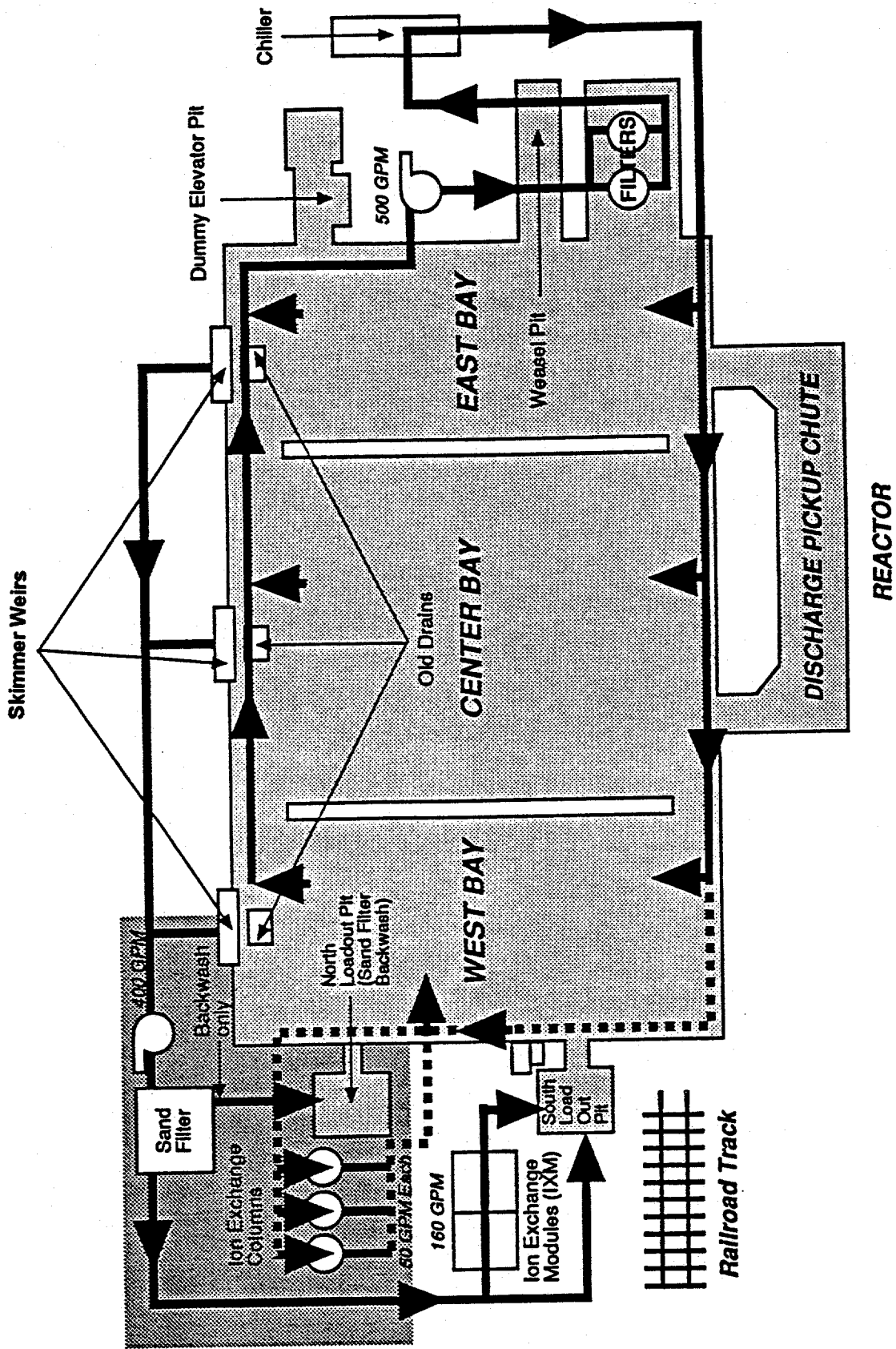
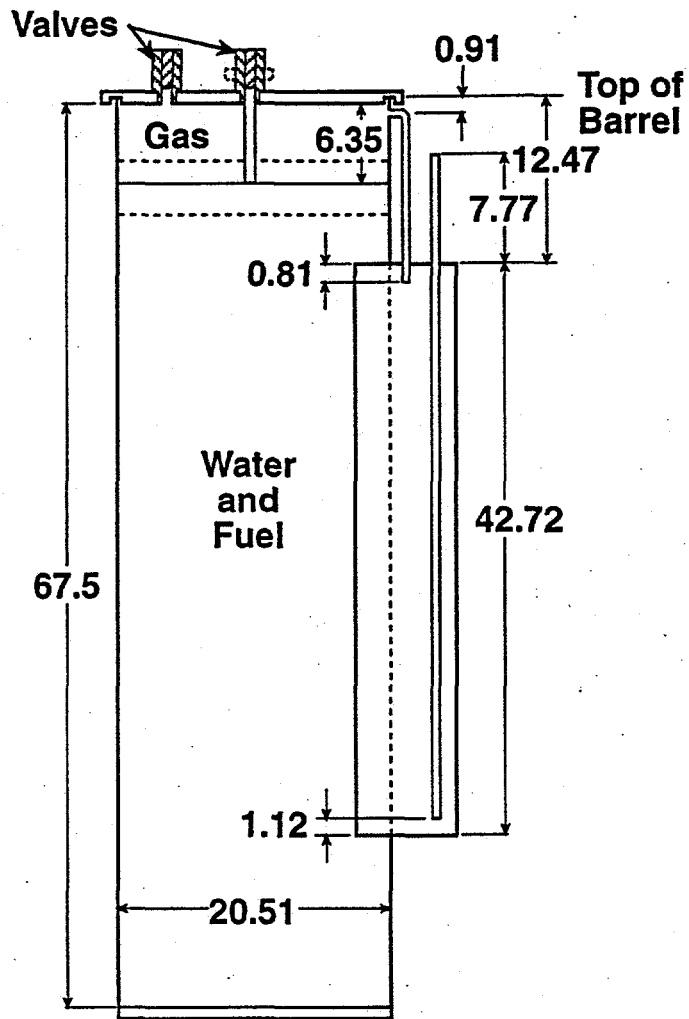


Figure 1-1. K-Basin Layout Map



Dimensions in cm

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Figure 1-2. Mk II Fuel Storage Canister Barrel

The water is passed through the sandfilter for the purpose of removing suspended particulates from the water. This filtration reduces the activity level of the water and prevents suspended particles from damaging the IXMs. Since the activity level of the water is reduced in this process, the concentration of radionuclides released into the air, in or near the basins, is also reduced.

The sandfilter is normally backwashed every three months to improve the water filtration flow-rate capacity. The water from the backwash is discharged to the north loadout pit, which is isolated from the rest of the basin. Routine monitoring is performed to determine radiological constituent activity levels in the water exiting from the IXCs and IXMs to assure that these are operating efficiently. This monitoring permits estimation of the build up of radionuclides in the components of the filtration system.

1.1.3 Fuel Storage Canisters

Spent fuel stored in the 105-KW Basin is contained in closed canisters. Each canister consists of two barrels, and each barrel contains up to seven N-Reactor fuel assemblies (Hanson, 1980, H-1-43445). The barrels are attached but independent. Two canister designs are used, MK I and MK II. They are similar in design with primary differences in the way the lid is sealed to the barrel and the configuration of the gas trap. A representation of a MK II canister barrel is shown in Figure 1-2. The canisters are designed to isolate the water in canister barrels from the basin water, while accommodating basin thermal cycles and venting of excess gas generated by fuel corrosion.

The fuel was loaded into the canisters in the 105-N Basin, the lid installed, potassium nitrite added, and a nitrogen gas space established before transporting to the 105-KW basin for storage. A gas space was established by purging nitrogen through the canister barrel from the off-center lid valve. This flushed water from the top of the barrel and the gas trap thereby isolating the canister water from the basin. A gas space of 6.4 cm (2.5 in) was assured by purging until gas bubbles were emitted from the open center valve before the valves were closed. Barrel lids which did not seal were replaced. Transporting the canisters from the 105-N Basin to the 105-KW Basin cycled the barrel gas pressure causing the gas trap to be partially filled with water (Conn 1992a, Conn 1992b, Conn 1993). However, filling of the gas traps with water should not reduce the effectiveness of the isolation of the barrel contents from the basin water.

1.2 SUMMARY OF EXISTING CONDITIONS

This SAP applies only to the 105-KW Basin which currently contains 3,821 sealed (MK I and II) canisters filled primarily with N Reactor fuel. Of the 3,821 canisters, 1,773 are the MK I variety that include 777 aluminum and 996 stainless steel canisters. All of the MK II canisters are constructed of stainless steel. The MK I and II canisters provide the primary barrier for the fission products escaping from the damaged fuel assemblies. The basin cooling water provides a secondary barrier to the potential release of radioactive materials.

A program is currently underway (Fulton 1994) to investigate alternatives for upgrading the fuel containers, removing the fuel from the vicinity of the Columbia River, and eventually, permanently disposing of the fuel. Proper evaluation of the alternatives requires information

about the state and condition of the fuel elements in the canisters. These data will be obtained from laboratory analyses of selected fuel elements (Abrefah, et al. 1994) which have been removed from the 105-KW basin. The sampling and analysis covered by this SAP supports the selection of the fuel elements to be examined.

The fuel elements that are candidates for characterization have been narrowed to those stored in the sealed MK II canisters. Some of these canisters contain broken fuel elements which have exposed the metallic uranium to the canister water. The exposed uranium surfaces have reacted with the water, releasing fission product nuclides to the canister water. The extent of this release is unknown, but large radionuclide inventories could cause significant releases to the basin water when the canister barrel lid is removed. To avoid such releases, samples of the liquid (and gas) in the canisters will be obtained from valves located on the top of each canister barrel. These samples will be evaluated for fission product content before the canister barrel seals are broken and their contents are exposed to the basin water.

Fuel cladding damage occurring during reactor discharge or subsequent handling exposes metallic uranium to water. The uranium will react with the water (corrode) forming hydrogen gas and releasing fission product nuclides. Gasses may also be produced by radiolytic decomposition of the water. Gasses formed in a canister barrel will combine with the nitrogen purge gas and excess pressure of the mixture will escape into the gas trap maintaining a constant pressure in the barrel consistent with the pressure of the water at the bottom of the basin. As the gas trap fills with gasses from the barrel, water in the trap is pushed out. When the gas trap water level is lowered to the bottom of the gas trap tube (1.12 cm (0.44 in) from the bottom of the trap), gas will be emitted to the basin.

As uranium is corroded consuming water, the water level in the barrel will drop below the 6.4 cm (2.5 in) tube (lid stem) that had established the 6.4 cm (2.5 in) gas space during the nitrogen purging of the barrel. Fission gasses in the fuel (such as ^{85}Kr) will be released as the uranium corrodes, and these gasses will be added to the hydrogen-nitrogen mixture in the gas space. Water soluble fission nuclides will also be released combining with the canister barrel water. The primary water soluble nuclide is ^{137}Cs . Smaller amounts of ^{90}Sr and ^{134}Cs are also expected.

Prior to the sampling required under this SAP the fuel canisters to be examined will have been moved to the Weasel Pit.

1.3 PURPOSE AND SCOPE

The purpose of the data obtained from the sampling and analyses described in this SAP is two fold: to estimate the radiological impact on the basin environment if the canister lids were to be removed, and to obtain data that will aid in characterizing the contents of the canisters for a variety of purposes.

Initially a determination of the ^{137}Cs concentration will be made by a mobile laboratory to guide decisions about opening canister lids for the purpose of removing fuel elements for hot-cell characterization analyses. If the radiological impact of removing the lid may be too large, the

canister barrel will not be opened. The concentration of ^{137}Cs that would prevent removing the lids will be provided in the Master Work Plan (MWP 1995) covering these activities. The mobile laboratory will also attempt to determine the amount of ^{85}Kr in the samples because significant quantities of this isotope could create radiological problems.

The broader purpose of these SAP activities is to obtain subsequent data that will aid in characterizing the contents of the canisters for a variety of purposes. The analytes of interest in both the liquid and gas samples are:

Liquid Samples

Cesium (^{137}Cs)
 Potassium, Nitrites, Ammonia, Nitrates
 Strontium (^{90}Sr)
 Plutonium (^{238}Pu , $^{239/240}\text{Pu}$)
 Uranium
 Tritium (^3H)
 pH of the liquid

Gas Samples

Hydrogen
 Argon
 Nitrogen
 Oxygen
 Fission Gas (All detectable isotopes of Kr, Xe)

The gasses in the gas samples (H, Ar, N, O) will be used to estimate the loss of the original cover gas, hydrogen production from fuel element corrosion, and the amount of radiolysis that may have taken place. The fission gas will give an indication of the amount of corrosion of the fuel elements.

The amount of corrosion will also be estimated from the ^{137}Cs content in the liquid samples. The pH of the liquid samples and the amount of nitrates, nitrites, and ammonia will determine the amount of corrosion inhibitor left in the canisters and thus, its effectiveness.

The Test Engineer shall be responsible for determining and recording the type of the samples (i.e. obtained liquid when gas was expected or gas when liquid expected). After the mobile laboratory analyses are complete the liquid samples will be shipped to the Westinghouse Hanford Company (WHC) 222-S Laboratory for analysis. It is expected that the gas samples will be sent to a contractor laboratory. A discussion of the analytical methods to be used in this sampling plan may be found in Section 3.0 .

The scope of this SAP is to provide a complete description of the procedures and the organizations responsible for performing those procedures that will ensure that the final laboratory results obtained meet the objectives of this activity and are of the required quality. The specific procedural areas addressed are summarily itemized in the Table of Contents of this document. The required organizations and their responsibilities are discussed throughout the text and summarized in Section 9.0.

The mobile laboratory will attempt to determine the amount of ^{85}Kr in the samples. No accuracy or limits are specified for this isotope in Section 3.0 because it is not currently known if the quantity of that isotope can be extracted from the gamma-ray spectrum.

1.4 DATA QUALITY OBJECTIVES

The DQO process was used in the development of this sampling program. This process included the following areas.

- The decisions to be made based on the data. These decisions answer the following questions.
 - Should the sealed canister barrel be opened at the basin(i.e. lid removed)?
 - What are the fission product problems for interim storage and processing?
 - What has been the performance of the corrosion inhibitors used?
 - How much radiolysis gas has been produced in wet storage?
 - Can this limited screening method replace more costly hot-cell examinations?
- The data to be acquired and the techniques to be utilized. The sampling techniques and the analytes required to make each of the decision above were examined.
- Sampling boundaries. Sampling and analysis will include an initial period where the sampling equipment and the associated sample analysis will be demonstrated. The extent of the final, production run has not been established.
- Decision logic. The manner in which the data acquired will be used to make the decisions described above.
- Decision errors. The risks of failure to obtain the data or of obtaining an incorrect result were examined.
- Decision optimization. The possible results that experience with this SAP may produce were examined to determine how they may impact optimization of the experimental program.

The objectives of the sampling program are presented in Section 1.3, while details on the sampling locations and frequency can be found in Section 2.1. The proposed analytical procedures, along with the required precision, accuracy, and practical quantification limit (PQL) requirements are outlined in Section 3.0. Additional details on records management and QA methods and procedures are addressed in Sections 4.0 and 5.0.

Reference has been made to WHC approved Standard Operating Procedures (SOPs) for specific sample collection procedures. Further details on the sampling and measurement approach are presented in Section 2.0.

The data collected under this SAP to determine the acceptability of opening canisters or of transporting sample vials will have definite limits specified in the Master Work Plan (MWP 1995). These limits are appropriate if the measurements are accurate to $\pm 10\%$. Therefore the precision and accuracy requirements for the mobile laboratory analyses must be 10% (two standard deviations). The minimum detection limit for normal ^{137}Cs data is $0.1 \mu\text{Ci}$ because this is one tenth of the lowest activity of concern.

The uncertainty in how well the results from the liquid and gas samples can determine the amount of corrosion and radiolysis has taken place, the effectiveness of the corrosion inhibitor that was originally in the canisters and the amount of fission gas that will be released when the canisters are opened can not be accurately established at this time. Consultation with the affected laboratories indicate that the constituents of the liquid and gas samples can be determined to within 10 to 25% using reasonable cost techniques. These values (two standard deviations) will be used as the liquid and gas sample requirements until subsequent analyses using the measurement results indicate that different limits are required or are permissible. The minimum detection limits estimated by the laboratories (and shown in the tables of Section 3.0) will similarly be used as requirements.

2.0 SAMPLING AND MEASUREMENT APPROACH

The following sampling and measurement procedures will be followed to ensure that the required data is collected and analyzed to support the purposes given in Section 1.3.

2.1 TYPE/FREQUENCY

All of the samples obtained under this SAP will be collected in 15 mL sample vials (See Section 2.2). They will be classified as liquid (>10 g net sample weight), non-liquid (>3 g, <10 g), or gas (<3 g). Samples will be obtained from each barrel (total of two) of each selected canister.

The number of samples to be taken from each canister barrel may vary as discussed in Trimble, 1995. If the barrel contains sufficient gas a gas sample from either the center or side valves on the top of the barrel will be obtained. A liquid sample will be obtained from the center valve. It may be necessary to flood the barrel to raise the liquid level high enough however. As many as two samples per barrel may be collected that do not meet either the liquid or gas criteria. These will be analyzed as liquid samples in the laboratories.

2.2 SAMPLING EQUIPMENT AND PROCEDURES

All of the samples collected under this SAP will be obtained using the sampling device designed for this purpose (Pitkoff 1994a, Pitkoff 1994b). The steps that K-Basin operators will take to obtain the samples (and open the valves on the top of the canisters, etc.) using this device are contained in Operating Procedure 60-43-12.

2.3 SAMPLE LABELING AND HANDLING

All samples collected as part of this sampling program shall be controlled as required by WHC-CM-7-7, EII 5.1, *Chain of Custody/Sample Analysis Request*. The EII 5.1 requirements apply as soon as the sample material is introduced into the sample container. Particular care must

be taken to assure that there are no periods on the Chain of Custody (COC) form with no custodian.

A field notebook (WHC-CM-3-5, Section 12.8) will be supplied by Nuclear Fuel Evaluations (NFE) to record all unusual or noteworthy observations made during the sampling process. Any participant may make entries in this notebook. When making corrections in the notebook (or on the COC) a single line shall be used to line out the original entry. The corrected entry shall then be made and shall be initialed and dated by the recorder.

All of the samples will be collected in 15 mL vials. These vials will be provided by Process Systems (PS).

NFE will mark each vial with a permanent, unique identification number and create a COC. This form, Figure 2-1, will include the vial identification number and the tare weight in grams. The tare weight will be obtained by the mobile laboratory (See Section 9.3.1) and will include not only the evacuated sample vial but the bags and ties used to contain the sample.

The samples will be collected by K Basins Operations (KBO). NFE will initiate the COC and record the following on the COC and the canister data sheet contained in the Master Work Plan (MWP 1995).

- The unique sample identification number
- The tare weight of the sample vial prior to sampling
- The date and time the sample was collected
- The canister number, barrel (marked or unmarked), and valve (center or side) from which the sample was collected.

If the sample yields too high a radiation level on contact (determined by K Basins Radiological Control (KBRC)) no further analysis of the sample will take place. The KBRC radiation results will be recorded by NFE on the COC and the sample will be returned to the basin for long term storage. This disposition of the sample will be noted by NFE on the COC. The COC will be retained by KBO and a copy of the COC and the MWP data sheet will be retained by NFE as permanent records. The criteria for acceptable radiation levels will be provided in the MWP.

The samples will then be transported to the mobile laboratory where they will be weighted. The gross weight of the sample vial (including the bags and ties) will be recorded on the COC by NFE. NFE will compute the net sample weight by subtracting the tare weight from the gross weight. This value will be corrected (reduced) by the calculated amount of sample line flushing liquid picked up with the sample and the basin water trapped between the outer sample sleeve and the vial. NFE will then record the corrected net sample weight on the MWP data sheet.

If directed by NFE, and recorded on the COC, the mobile laboratory will also measure the curies of ^{137}Cs (and ^{85}Kr if possible) contained in the sample vial. This information, in units of mCi, will be recorded on the COC form and the MWP data sheet (^{137}Cs only) by NFE.

NFE will then determine the type of subsequent analysis the sample should undergo (liquid or gas) and the out-of-area laboratory that should perform it, (WHC 222-S or Contract, See Section 9.3) and record this on the COC form. The designations "liquid" or "gas" will be sufficient to indicate the analytical procedures to be used by the laboratory. If the sample exhibits too high a radiation level (criteria given in the MWP) no further analyses may be performed and the sample may be returned to the basin. The actions and retained records are as described above for samples returned to the basin.

KBO will then prepare the samples to be shipped to the out-of-area laboratories. It is the intent of this SAP to ship the samples to the laboratory within four days of the day they are collected. If logistics preclude shipment the day the sample is collected, it shall be stored in an area away from any sources of contamination when not in the possession of the sampler. Samples will be shipped out of the K area by a WHC designated shipper. The samples will be transported in either the PAS-1 cask, a DOT 7A container, or NFE approved alternates.

A copy of the COC form completed by the receiving laboratory will be provided to NFE by the shipper after the transfer is complete.

2.4 SAMPLE PRESERVATION AND HOLDING TIMES

The samples being collected for analysis of the liquid have no sample preservation requirements. There are also no requirements for preservation of the gas samples, however, the gas samples should not be exposed unnecessarily to temperatures higher than 43°C (110°F) to minimize the loss of hydrogen.

The phrase "holding time" refers to the allowable time lapse between sample collection and laboratory analysis. If an analysis is performed after the holding time is exceeded, the analytical results must be considered invalid. The holding time limit on the analysis of the liquid samples is 60 days due to possible irradiation effects on the sample vial materials. The gas samples have holding times of 15 days because of the potential loss of hydrogen.

The pressure inside the sample vials could have an impact on the allowable holding time. The pressure inside the sample vials will be approximately 0.5 atm gage when they are brought to the surface of the basin. Laboratory tests show that the stoppers sealing the vials will contain the samples up to a pressure of 1.0 atm gage. Tests have been initiated to verify that the stoppers can continue to hold such pressures over time. Six test samples with internal gage pressures of 0.6 to 0.9 atm and temperatures of 21 to 43°C (70 to 110°F) have been created. These test vials will be monitored on a daily basis. If the stoppers fail, the above holding times will be immediately modified. It should be noted that failure of a stopper does not constitute a radiological or safety problem. The energetics will not break the double bagging surrounding each vial.

3.0 LABORATORY ANALYSIS OF SAMPLES

The analyses, analytical limits, quality assurance samples, and reporting requirements for the laboratories (described in Section 9.3) participating in this SAP vary noticeably. Therefore, each of the above subjects is addressed below by laboratory. The definition of the parameters used to quantify the quality assurance limits placed on the analyses, however, are common to all and are listed below.

Precision Precision represents a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is calculated by the laboratory from the analytical results from three triplicate samples obtained by splitting a single field sample into three or more parts. The precision of these three analyses is twice the standard deviation of the results divided by their mean. This parameter quantifies the repeatability of laboratory analyses. (Precision including sampling variability are discussed in Section 5.0)

Accuracy Accuracy represents the degree of agreement of a measurement with an accepted reference or true value. Sample accuracy is calculated by the laboratory based on average percent recoveries of spiked samples. This parameter is only meaningful for the liquid sample analyses performed by the 222-S laboratory.

Practical Quantification Limits (PQL) PQL represents the minimum sensitivity the analytical procedure must meet in order to satisfy the data requirements. It is an absolute quantity (e.g. Ci/g). Analyte concentrations below this value are of no concern to the DQOs.

Minimum Detection Level (MDL) MDL represents the minimum level of the analyte that can be detected by the analytical procedure. PQL must be greater than MDL.

For all laboratory reports, the actual analysis results shall be reported for values less than the PQL but greater than the MDL (i.e. $< \text{PQL}$ should not be used to represent the result).

The laboratory procedures specified in the following tables are the ones currently in use. Revised procedures that improve or correct the specified ones may be used if the ability of the analytical results to meet the specified PQL, precision, and accuracy requirements is not impaired.

3.1 MOBILE LABORATORY

The mobile laboratory will perform gamma energy analyses (GEA) to determine the ^{137}Cs activity of liquid samples. The PQL, precision, and accuracy requirement for this analysis are given in Table 3-1. The activity of ^{85}Kr will also be determined if possible (See Section 1.3) but no requirements are placed on this assessment.

The PQL and accuracy requirements will be verified with a standard sample supplied by the laboratory. This standard will also be used to calibrate the counting/sample configuration.

This calibration shall be repeated after each precision determination (described below). The accuracy of the standard will determine the accuracy of the counting system.

The precision of the sample mounting and counting system and procedures will be assessed for 10% of the samples by remounting and counting a sample two additional times. The standard deviation of the results must be 1/2 of the required precision.

Table 3-1. Analytical Procedures and Process Requirements for Mobile Laboratory Liquid Samples

Process	Constituents	Procedure ²	Required PQL	Required	
				Precision	Accuracy
Weight	Weight of Vial	DI ²	0.05 g	±0.01 g	±0.01 g
GEA	¹³⁷ Cs	DI ²	0.1 µCi	±10%	±10%
GEA	⁸⁵ Kr ¹	DI ²	10 µCi	±10%	±10%

¹ The ⁸⁵Kr parameters are goals not requirements.

² The procedures to be followed are contained in a Desk Instruction (DI) titled, *Desk Instruction for KW-Basin On-Site Sample Analysis*. This DI and any revisions to it will be reviewed and approved by NFE.

The total ¹³⁷Cs (and ⁸⁵Kr if available) activity in units of mCi, the counting statistics uncertainty, and the gross weight of each sample will be reported to NFE as soon as they are obtained. These data will be reported on a laboratory created form that includes the vial identification number and the tare weight obtained prior to sampling. Data records containing the total counts obtained for each sample, the magnitude of the standard, the counts obtained for each calibration run and each precision run shall be maintained and made available for NFE review. A final written report summarizing these data records will be completed and forwarded to NFE at the completion of the program.

3.2 CONTRACTOR LABORATORY

The contractor laboratory will analyze gas samples for the constituents given in Section 1.4 using a mass spectrometry technique. The analytical procedure and PQL, precision, and accuracy requirements for this analysis are given in Table 3-2.

The precision of the measurement system and procedures will be assessed for 10% of the sample vials by extracting two additional analytical samples from the vial. The standard deviation of the results must be 1/2 of the required precision. Note that this requirement only applies for constituents which have an abundance greater than 0.5% of the total mass in the analytical sample.

**Table 3-2. Analytical Procedures and Process Requirements
for Contract Laboratory Gas Samples**

Process	Constituents	Procedure ID	Required PQL	Required	
				Precision	Accuracy
Mass Spectroscopy	H, Ar, N, O, Kr, Xe	PNL ALO 284, Rev 1	20 ppm	±10%	±10%

The PQL and accuracy requirements will be verified daily with a pure nitrogen calibration standard supplied by the laboratory. The accuracy of the recovery of the nitrogen in the standard will determine the accuracy of the measurement system.

The results will be reported as the volume % of the constituents of the sample withdrawn from the vial.

The laboratory will submit a final report for each shipment of samples within 45 calendar days after receipt of the samples. Interim preliminary data reports will be submitted to NFE as requested. The final report shall include:

- A copy of the COC form
- Laboratory identifiers cross-referenced to vial identification numbers
- Dates of laboratory sample extraction and analysis
- The analytical results as indicated above
- Identification of data outliers or deficiencies
- Results from laboratory standards
- Laboratory procedures used (documentation reference) and any deviations from those procedures.
- Accuracy and precision determinations made at the laboratory
- Detection limits and their basis.

The individual final reports will be combined by NFE to create a project data package.

3.3 222-S LABORATORY

The 222-S laboratory will analyze liquid samples for the constituents given in Section 1.3 using a variety of analytical techniques. The analytical procedures and PQL, precision, and accuracy requirements for this analysis are given in Table 3-3.

The precision of the measurement system and procedures will be assessed for at least 10% of the sample vials by extracting two additional analytical samples from the vial. The standard deviation of the results must be 1/2 of the required precision

The PQL and accuracy requirements will be verified with matrix spikes (See Section 5.2) supplied by the laboratory. Matrix spikes are not required for the Sr, Pu, and U analyses because tracers are used in the analytical procedure. The accuracy of the recovery of the spikes and tracers will determine the accuracy of the measurement system. These accuracy assessments must be made after each precision assessment.

The results will be reported as the total volume (mL), total mass (mg) and constituent densities (See Table 3-3 for units) of the sample withdrawn from each vial.

The laboratory will submit a final report for each shipment of samples within 90 calendar days after receipt of the samples. Interim preliminary data reports will be submitted to NFE as requested. The data in the interim reports will be verified by an internal technical review, as evidenced by a signature on the reports. The final report shall include:

- A copy of the COC form
- Laboratory identifiers cross-referenced to vial identification numbers
- Dates of laboratory sample extraction and analysis
- The analytical results as indicated above
- Identification of data outliers or deficiencies
- Results from matrix spikes, tracer recoveries, laboratory duplicates, and any other laboratory control samples
- Laboratory procedures used (documentation reference) and any deviations from those procedures.
- Accuracy and precision determinations made at the laboratory
- Detection limits and their basis.

The individual final reports will be combined by NFE to create a project data package.

**Table 3-3. Analytical Procedures and Process Requirements
for 222-S Laboratory Liquid Samples**

Process	Constituents	Procedure ID	Required PQL	Required	
				Precision	Accuracy
Sample Dissolution	Pu, and U	LA-505-159	NA	NA	NA
Separation	Pu	LA-503-156	NA	NA	NA
Mounting	Pu	LA-542-101	NA	NA	NA
AEA	²³⁸ Pu, ^{239/240} Pu	LA-508-161	0.5 μ Ci/mL	$\pm 25\%$	$\pm 20\%$
Laser Fluorimetry	U Total	LA-925-009	10 μ g/mL	$\pm 20\%$	$\pm 20\%$
Sample Mount	Gamma Emitters	LA-548-121	NA	NA	NA
GEA	¹³⁷ Cs	LA-508-162	0.001 μ Ci/mL	$\pm 10\%$	$\pm 10\%$
GFPC	⁹⁰ Sr	LA-220-101	0.1 μ Ci/mL	20%	20%
L.S.	³ H	LA-218-114	0.1 μ Ci/mL	20%	20%
ISE	pH of Liquid	LA-212-102	NA	10%	10%
ICP	Potassium	LA-505-151	1 mg/L	20%	20%
ISE	Ammonia	LA-631-001	5 mg/L	20%	20%
IC	Nitrate, Nitrite	LA-533-105	1 mg/L	20%	20%

4.0 DATA MANAGEMENT AND RECORD KEEPING

The implementation of this SAP will generate sampling and analysis data. These data will be collected, analyzed, stored, and made readily retrievable by NFE. Currently, it is not planned to create a widely accessible database for these data because they are not of general concern throughout the site. Therefore, the provisions of WHC-CM-2-6 for data management programs generally do not apply. Proper Records Inventory and Disposition Schedules (RIDS) will be generated for the data files, however.

All of the generated data will be controlled as permanent project quality records, as required by WHC-CM-4-2, QR 17.0, *Quality Assurance Records*, WHC-CM-3-5, *Document Control and Records Management Manual*, or equivalent WHC approved procedures. This requirement also applies to the internal records created by each of the laboratories involved.

The data records that will be managed by NFE for this activity include:

- Sampling notebooks
- Copies of the Canister Data sheets from the MWP
- Copies of the COC forms
- Interim laboratory reports
- Final laboratory reports
- Data validation and verification reports

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

The sampling and analysis activities in this plan were developed to ensure that the DQOs of Section 1.4 are met and that the applicable QA requirements of WHC-CM-4-2, *Quality Assurance Manual*, are satisfied.

In addition, the 222-S Laboratory shall follow WHC-SD-CP-QAPJP-003, *Quality Assurance Program Plan for the Chemical Analysis of Environmental Samples*. MCS-033, *Quality Assurance Plan for Activities Conducted by the Analytical Chemistry Laboratory (ACL)* will be used if Pacific Northwest Laboratories (PNL) is selected as the contractor laboratory.

5.1 QUALITY ASSURANCE

The data will be considered representative so long as at least 90 percent of the data points meet the established requirements for precision and accuracy. During the data validation process (Section 5.3) data that do not meet this objective will be reviewed to determine whether they can be used or whether corrective action shall be taken. If necessary, this corrective action may consist of updating the sampling and/or analysis activities.

5.2 QUALITY CONTROL

The precision and accuracy of the analysis performed by each laboratory are routinely evaluated in accordance with the laboratory QA plans. Additional QC samples are required by this plan and are described below.

- **Triplicate Samples:** Each laboratory will prepare triplicate samples that will be carried through the extraction, preparation, and analysis steps. This will test the precision of the complete procedure at each laboratory, starting at the receipt of a canister sample. Since there is no extraction or sample preparation steps required at the mobile laboratory, a single sample vial will be mounted and counted three times to meet this requirement. At the 222-S and contractor laboratories two additional analytical samples will be withdrawn from the selected sample vial. Triplicate analyses shall be performed on a minimum of 10% of the samples.
- **Matrix Spike Samples:** Matrix spike samples will be prepared for all analytes that do not employ tracers or carriers in the analytical procedures. The samples, required only for the liquid sample analyses at the 222-S laboratory, require the addition of a known quantity of representative analytes to the sample to measure analytical accuracy. The spike samples shall be created from replicates of a normal sample. Matrix spike samples, when required, will be analyzed for each batch of samples processed.

Additional samples will be created in the field to verify the adequacy of the sampling and sample handling operations. These are described below.

- **Blind Samples:** At the direction of NFE, blind reference samples may be introduced into any sampling round as a performance assessment of the laboratory.
- **Field Blanks:** Field blanks consist of pure, deionized, distilled water, transferred into a sample container at the sampling site. Field blanks are used as a check on environmental contamination and the adequacy of sampling equipment decontamination procedures. They shall be collected at the same frequency as field duplicate samples.
- **Field Duplicate Samples:** A minimum of 5 percent of the samples collected shall be duplicates. The duplicate samples are second samples taken under the same conditions from the same canister barrel and valve. Variations in the results indicate the variability in the sampling (and analytical procedures). NFE will direct the collection of field duplicate samples.

5.3 DATA VERIFICATION AND VALIDATION

The data packages received from the laboratories shall be verified and validated by NFE prior to any official use of the data. The verification process will consist of reviewing each laboratory report to confirm that the sample number, sampling time and date, sampling location, and analytical methods performed are consistent with that requested by the COC form and this SAP. The process will also confirm that the laboratory report is complete and contains all of the elements listed in Section 3.0.

Data validation is a process used to check procedures and data for validity, completeness, and overall QA. The validation process will reflect the requirements set forth in the DQOs (DOE

1994). The information required for the final data reports (Section 3.0) will normally be sufficient for this process, however, NFE may make requests for additional information from a laboratory.

NFE shall evaluate and confirm the laboratory's assessment of accuracy, precision, and minimum detection limits using the matrix spike, laboratory standard, duplicate/triplicate sample, and blind field sample analytical results. The packages shall also be evaluated to meet the QA completeness and comparability requirements defined below.

Completeness Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. A complete data package is one that contains valid results (i.e. accuracy, precision) for all of the analytes specified in the DQOs. In some cases, data may not meet all the requirements but may still be used for qualitative information. NFE will determine the completeness of the data after the data validation is complete.

Comparability Comparability is the confidence with which one data set can be compared to another. Data for both the liquid and gas samples may be used to assess the same physical parameter of the canisters (e.g. corrosion). If the analyses meet their respective limits (Tables 3-1, 3-2, and 3-3), the assessments will be comparable. This parameter will be of concern only if alternative laboratories or analytical methods are required. Comparability with existing data will be evaluated if that situation occurs.

The final validation performed by NFE will be to assess the representativeness of the data, as defined below.

Representativeness Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, a process condition, or an environmental condition. Representativeness of a population or an environmental condition is heavily dependent on the sample collection method and strategy. Representativeness of the sample process condition and the methods shall be assessed by NFE after accumulation of sufficient data to represent the same population. Data representativeness is addressed qualitatively by the selection of the canisters to be sampled (Controlled by the MWP).

6.0 EQUIPMENT DECONTAMINATION

The sampling equipment will be decontaminated (purged) between each sample collection by flushing the injection housing for one minute with demineralized water (See Operating Procedure 60-43-12). Sample vials will not be decontaminated, rather only new, clean vials shall be used.

7.0 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

Investigation-derived waste expected to be derived from the proposed sampling program include disposable sampling units, decontamination fluids, paper towels, and disposable personal protective clothing such as gloves, disposable liners, and booties. Disposal of non-reusable items shall be done in accordance with HSRCM-1, *Radiological Control Manual* and standard radiological work procedures.

8.0 HEALTH AND SAFETY

Worker health and safety procedures that will be followed for sample collection activities are outlined in WHC-IP-0718 *Health Physics Procedures*, and HSRCM-1 *Hanford Site Radiological Control Manual*. All sampling procedures shall have the independent review and approval of the WHC Safety Assurance Organization.

9.0 RESPONSIBILITIES

The following sections outline the general responsibilities of the organizations responsible for performing the actions required by this SAP. Figure 9-1 shows the lines of authority for the collection and analysis of the samples and the organizational path of the sample vials.

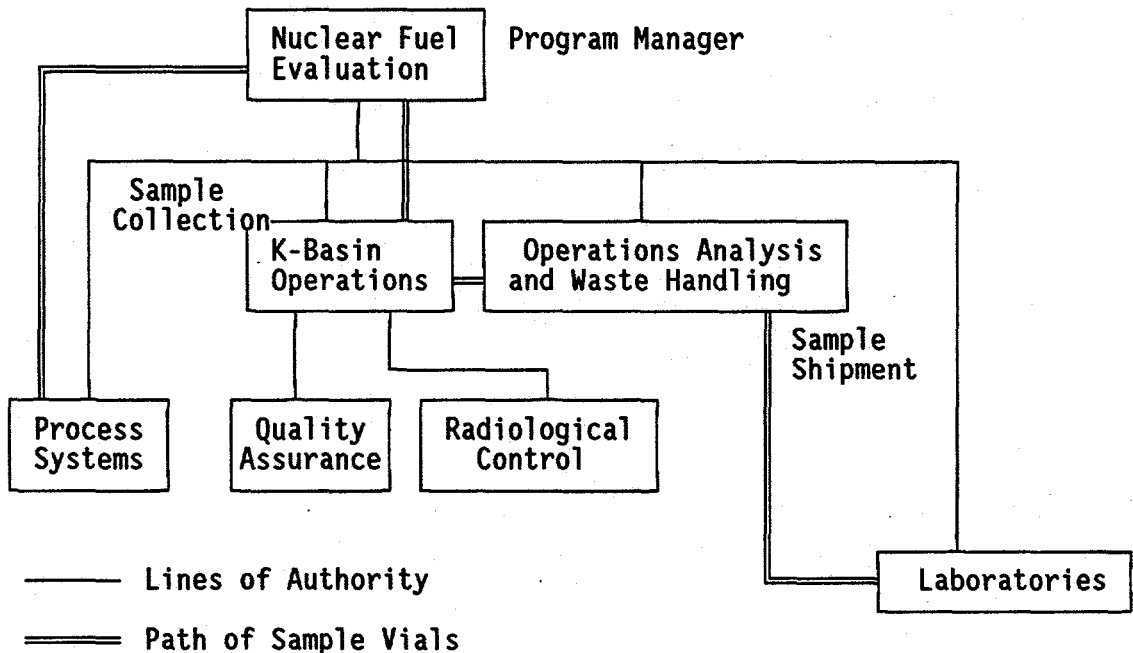


Figure 9-1. Program Lines of Authority

9.1 PROJECT MANAGEMENT

Activities in the Project Management Program include: preparing procedures, requesting samples and analyses, performing monitoring activities and obtaining samples, maintaining equipment, and data management. The organizations responsible for these activities are presented in the following sections.

9.1.1 Nuclear Fuel Evaluations

NFE is the Project Manager and is responsible for obtaining the required data and utilizing it to make the required decisions (under other SAPs and procedures). NFE responsibilities include the following:

- Providing an on-site NFE representative (Test Engineer)
- Specifying the samples to be taken on each canister.
- Determining the requirements for the use of Quality Control (QC) samples.
- Assigning sample ID numbers
- Labeling samples and completing label forms
- Initiating the COC form
- Recording data on the COC form and MWP data sheet
- Maintaining files of all sample information.
- Maintaining a database of relevant information.
- Performing data validation.
- Performing data management and record keeping.
- Providing mobile laboratory information to KBO and Operations Analysis and Waste Handling (OAWH).

9.1.2 K Basins Operations

KBO is responsible for operating and maintaining the K Basins in compliance with established safety and environmental requirements. KBO responsibilities include the following:

- Sampling and monitoring in accordance with all relevant SAPs
- Collecting, delivering samples to the mobile laboratory (Section 9.3.1) for analysis and preparing samples for shipment to laboratories outside of the K area.

- Training operators to perform the required activities in accordance with approved procedures
- Initiating corrective action and notifications when sample parameters exceed applicable levels or limits.
- Decontaminating sampling equipment, etc.

9.1.3 Operations Analysis and Waste Handling

OAWH is responsible for sample shipments out of K area. Their responsibilities include:

- Ensuring that correct COC procedures are followed when transferring sample custody.
- Packaging, shipping, and transporting all samples to the 222-S and Contractor Laboratories.

9.2 SUPPORTING ORGANIZATIONS

Other organizations that support the collection of samples from the K Basin include: Process Systems, K Basins Radiological Control (KBRC) and QA. The following section outlines the primary responsibilities of each of these organizations:

9.2.1 K Basins Radiological Control

KBRC, in direct support on the sampling and packaging required by this SAP is responsible for:

- Reviewing technical work documents (work packages and procedures) for inclusion of Radiological Control hold points and work instructions.
- Preparing Radiation Work Permits (RWPs).
- Providing Health Physics Technicians (HPTs) for job coverage.
- Enforcing requirements of the Hanford Site Radiological Control Manual (HSRCM-1, Rev. 2)

9.2.2 Quality Assurance

The primary responsibilities of QA include reviewing and approving changes and modifications to K Basin administrative control documents, performing periodic inspections to ensure that basin operations meet WHC administrative requirements, performing periodic appraisals and audits, and identifying training requirements for QA personnel supporting K Basin spent fuel storage basin operations.

9.2.3 Process Systems

PS provides the physical equipment needed to obtain the samples. Specifically, their responsibilities include:

- Providing the equipment to operate the valves on the barrels, collect the gas or liquid, inject the sample into the sample vials, and retrieve the sample vials.
- Providing all sample vials.
- Providing all spare parts for the equipment.
- Providing any auxiliary fixtures required by the sampling equipment.

9.3 ANALYTICAL LABORATORIES

Samples collected at K Basin shall only be analyzed by WHC operated laboratories or by a WHC approved participant contractor laboratory. For participant contractors, applicable quality requirements shall be included as part of the approved work order or procurement document. Laboratories must submit their analytical methods and internal QAPjP for WHC review and approval

9.3.1 Mobile Laboratory

A mobile laboratory facility from Special Analytical Studies/Hanford Technical Services will be set up at or in 105-KW to weigh the sample vials and to perform rapid gamma scan analysis of samples from the canisters. The analysis will be performed using the methods specified in Section 3.1.. Results will be prepared and sent to the on-site NFE representative.

9.3.2 222-S Laboratory

The 222-S Laboratory is expected to perform the majority of chemical and radiochemical analyses of the liquid samples collected with this SAP. The analyses performed shall be in accordance with the methods identified in Section 3.3. Laboratory results shall be sent to NFE who will review it to assure that it meets the minimal requirements requested.

9.3.3 Contract Laboratory

The Contract Laboratory, when requested by WHC, will perform chemical analysis of samples collected from K Basin as directed by NFE. They will primarily perform all of the analyses of the gas samples but may be requested (by NFE) to analyze some liquid samples. The analysis will be performed using the methods specified in Section 3.2. Laboratory results shall be sent to NFE who will review it to assure that it meets the minimal requirements requested.

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