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HANFORD PERFORMANCE EVALUATION PROGRAM FOR HANFORD
SITE ANALYTICAL SERVICES

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7. Abstract

The U.S. Department of Energy (DOE) Order 5700.6C, Quality Assurance, and Title 10 of the Code of Federal Regulations, Part 830.120, *Quality Assurance Requirements*, states that it is the responsibility of DOE contractors to ensure that "quality is achieved and maintained by those who have been assigned the responsibility for performing the work." Hanford Analytical Services Quality Assurance Plan (HASQAP) is designed to meet the needs of the Richland Operations Office (RL) for maintaining a consistent level of quality for the analytical chemistry services provided by contractor and commercial analytical laboratory operations. Therefore, services supporting Hanford environmental monitoring, environmental restoration, and waste management analytical services shall meet appropriate quality standards.

This performance evaluation program will monitor the quality standards of all analytical laboratories supporting the Hanford Site including on-site and off-site laboratories. The monitoring and evaluation of laboratory performance can be completed by the use of several tools. This program will discuss the tools that will be utilized for laboratory performance evaluations. Revision 0 will primarily focus on presently available programs using readily available performance evaluation materials provided by DOE, EPA or commercial sources. Discussion of project specific PE materials and evaluations will be described in section 9.0 and Appendix A.

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**HANFORD PERFORMANCE EVALUATION
PROGRAM
FOR HANFORD SITE
ANALYTICAL SERVICES**

September 1995

**Office of Quality Assessment
Analytical Services
Westinghouse Hanford Company
Richland, Washington 99352**

Hanford Performance Evaluation Program

September 1995

Approved By:

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LIST OF TERMS

AA	Atomic absorption
AEQA	Analytical Environmental Quality Assurance
AIHA	American Industrial Hygiene Association
ACL	Analytical Chemistry Laboratory
AO	Analytical Operations
APG	Applied Products Group
AS	Analytical Services
CA	Corrective action
CAA	Clean Air Act
CERCLA	<i>Comprehensive Environmental Response, Compensation, and Liability Act</i>
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
CP	Chemical Processing
CVAA	Cold vapor atomic absorption
CWA	Clean Water Act
DOE	U.S. Department of Energy
DOE/ID	U.S. Department of Energy Idaho Operations Office
DOE/RL	U.S. Department of Energy Richland Operations Office
DSC	Differential Scanning Colorimetry
ELPAT	Environmental Lead Proficiency Analytical Testing
EM	Environmental Management
EML	Environmental Measurements Laboratory
EMSL-CI	Environmental Monitoring Systems Laboratory, Cincinnati
EMSL-LV	Environmental Monitoring Systems Laboratory, Las Vegas
EPA	U.S. Environmental Protection Agency
ER	Environmental Restoration
ERA	Environmental Resource Associates
FY	Fiscal year
GC	Gas chromatography
GCMS	Gas chromatography mass spectrometry
GEA	Gamma energy analysis
GFAA	Graphite furnace atomic absorption spectrometry
GOCO	Government-owned contractor-operated
HASQAP	<i>Hanford Analytical Services Quality Assurance Plan</i>
HATS	Hanford Action Tracking System
HEIS	Hanford Environmental Information System
HPEP	Hanford Performance Evaluation Program
HPLC	High-performance liquid chromatography
HYAA	Hydride atomic absorption spectrometry
IC	Ion chromatography
ICP	Inductively coupled plasma spectrometer
ICP/MS	Inductively coupled plasma spectrometer/mass spectrometer
INEL	Idaho National Engineering Laboratory
IRM	Information Resource Management
ISE	Ion selective electrode
LABQAP	<i>Laboratory Quality Assurance Plan</i>
LCS	Laboratory control sample
LIMS	Laboratory Information Management System
LQAO	Laboratory Quality Assurance Officer
MAPEP	Mixed Analyte Performance Evaluation Program

MDA	Minimum detectable activity
MDL	Method detection limit
mRPD	Mean relative percent difference
NIST	National Institute of Standards and Technology
NESHAP	<i>National Emission Standards for Hazardous Air Pollutants</i>
NVLAP	National Voluntary Laboratory Accreditation Program
OQA	Office of Quality Assessment
PAT	Proficiency Analytical Testing
PCB	Polychlorinated biphenyl
PE	Performance evaluation
PNL	Pacific Northwest Laboratories
QA	Quality assurance
QAO	Quality Assurance Officer
QAP	Quality Assessment Program
QB	Quarterly blind
QC	Quality control
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QES	Quanterra Environmental Services
R	Recovery
RCRA	<i>Resource Conservation and Recovery Act</i>
RESL	Radiological and Environmental Sciences Laboratory
RIDS	Records Inventory and Disposition Schedules
RIS	Radiological Intercomparison Study
RSA	Request for Special Analysis
SAP	Sample Analysis Plan
SD	Supporting document
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SOP	Standard Operating Procedure
SOW	Statement of Work
SVOC	Semi-volatile organic compound
TAL	Target Analyte List
TC	Total Carbon
TCL	Target Compound List
TGA	Thermogravimetric analysis
TIC	Total inorganic carbon
TMA	Thermal Analytical Laboratory
TOC	Total organic carbon
TPA	Tri-Party Agreement (<i>Hanford Federal Facility Agreement and Consent Order</i>)
TWAP	Tank Waste Analysis Plan
TWRS	Tank Waste Remediation Systems
USGS	U.S. Geological Survey
VOC	Volatile organic compound
VTSR	Verified time of sample receipt
WA	Washington State
WAC	Washington State Administrative Code
WAP	Waste Analysis Plan
WDOE	Washington State Department of Ecology
WHC	Westinghouse Hanford Company
WM	Waste Management
WP	Water pollution
WS	Water supply
WSCF	Waste Sampling and Characterization Facility

1.0 INTRODUCTION

The U.S. Department of Energy (DOE) Order 5700.6C, *Quality Assurance*, and Title 10 of the Code of Federal Regulations, Part 830.120, *Quality Assurance Requirements*, states that it is the responsibility of DOE contractors to ensure that "quality is achieved and maintained by those who have been assigned the responsibility for performing the work." Hanford Analytical Services Quality Assurance Plan (HASQAP) is designed to meet the needs of the Richland Operations Office (RL) for maintaining a consistent level of quality for the analytical chemistry services provided by contractor and commercial analytical laboratory operations. Therefore, analytical services supporting the Hanford Site environmental monitoring, environmental restoration, and waste management analytical programs shall meet appropriate quality standards.

This performance evaluation program will monitor the quality standards of all analytical laboratories supporting the Hanford Site including on-site and off-site laboratories. The monitoring and evaluation of laboratory performance can be completed by the use of several tools. This program will discuss the tools that will be utilized for laboratory performance evaluations. This document will primarily focus on presently available programs using readily available performance evaluation materials provided by DOE, the Environmental Protection Agency (EPA) or commercial sources. Discussions on project specific performance evaluation (PE) materials and evaluations will be described in Section 9.0 and Appendix A.

1.1 Objective

Performance evaluation (PE) materials or samples are a necessary and required element of quality assurance and quality control (QA/QC) programs covering environmental analyses. Regulatory drivers and DOE orders mandate laboratory participation in specific PE programs. The performance sample provides an external and independent reference for monitoring individual environmental laboratory analytical programs. A PE program provides an evaluation of a laboratory's capabilities for analyzing specific components of known concentration in a particular sample matrix. Acceptable performance by a laboratory on PE material indicates that the laboratory's policies and procedures used for analyzing that material are satisfactory and that the laboratory is capable of performing these types of analyses. This external evaluation of the laboratory performance enhances the credibility of the laboratory and the overall confidence in the results it produces.

The Hanford Performance Evaluation Program (HPEP) was established to provide a central point of coordination and control on external and internal programs, evaluate laboratory qualification and to examine and analyze various samples from the Hanford Site.

The degree of independence required for a PE program depends on how the data is going to be used. If the data that the laboratory produces is being used to demonstrate regulatory compliance or some other application, where it may be reviewed for litigation or other external purposes, then the customer needs the highest level of confidence that the data is accurate. For these cases, a high level of independence is important to ensure the PE program's credibility. On the other hand, if the data is only being used internally to ensure that the waste composition is within specified operating limits for

safety or future processing, then the level of confidence and independence in the PE program is not as critical. PE's with a high degree of independence are normally more expensive to administer. They should be defined as part of the customers analysis requirements. Normal operations will include other analyses as part of its normal QA/QC program, which will provide internal performance evaluation information.

The PE material should be the same type of matrix as the sample, if the performance on the PE is going to be indicative of the performance of the laboratory for the analyses of the samples. Because no commercial or regulatory PE materials exist that are representative of the highly radioactive, high concentrated salt and sludge matrices of Hanford's Tank Waste Remediation System (TWRS) wastes, alternative approaches to performance evaluation materials are needed. The HPEP will include the present performance evaluation programs, the Sample Exchange/Evaluation (SEE) Program and future alternatives (section 9.1 and Appendix A).

1.2 Scope

The requirement for participation in the HPEP applies to all Hanford Site support service contractors performing analytical laboratory tasks. The HPEP addresses the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the Safe Drinking Water Act (SDWA), the Clean Water Act (CWA), the Resource Conservation and Recovery Act (RCRA), the Clean Air Act (CAA), and the American Industrial Hygiene Association (AIHA) analytical services. Contracted analytical service suppliers supporting customers and programs across the Hanford Site are subject to the requirements defined in this document.

1.3 Program Administration

The HPEP, ie., implementation of HPEP, is the responsibility of the Analytical Services, Office of Quality Assessment (OQA). OQA is a Quality organization (independent of the laboratories) reporting to the Analytical Services manager. Contract laboratory QA/QC staff, Quality Assurance staff, and technical specialist will be designated to provide support for the HPEP. The requirements for HPEP participation will be introduced, implemented, and monitored by OQA and contract laboratory designated staff.

2.0 ORGANIZATION AND PERSONNEL RESPONSIBILITIES

Functional responsibilities for HPEP are described in this section. An official listing of key personnel shall be maintained in the OQA Manager charter files and updated as changes occur.

2.1 HPEP Administrator/Manager

The OQA Manager will oversee the HPEP and manage the operational aspects. The OQA manager will ensure that all quality requirements of this document are reviewed annually and updated as appropriate. The OQA manager will identify and report any quality related problems, evaluate the availability of necessary personnel, equipment, and services needed to support the HPEP. The OQA manager will interface with participating laboratories and programs during the development of new PE programs and during problem resolution.

2.2 HPEP Quality Assurance (QA) Officer

The PE Program QA Officer is responsible for ensuring that all quality requirements of this program are met in this document. The PE Program QA Officer shall provide routine guidance on quality-related matters to the program staff. The PE Program QA Officer shall identify and report quality related problems within the PE Program to the HPEP Administrator/Manager, and shall initiate, recommend, and track associated corrective actions to closure. The PE Program QA Officer also reviews instances of supplier nonconformance and any associated corrective actions. The HPEP QA Officer will be responsible for assisting the HPEP Administrator/Manager in problem resolution.

2.3 HPEP Data Management Coordinator

The PE Program Data Management Coordinator is responsible for maintaining all HPEP performance indicators and performance status databases. The PE Program Data Management Coordinator is responsible for quarterly reports summarizing performance indicators and performance status of Hanford site contract and commercial laboratories to the HPEP Administrator/Manager, Analytical Services Management and RL.

2.4 Special Project PE Coordinators

The special project PE coordinators are responsible for coordination of the development, implementation and monitoring of special project PE programs. These coordinators are responsible for all sampling and testing requirements associated with the associated special project. These coordinators interface with the laboratory QA/QC Officers during the scheduling and tracking of analytical activities. The coordinators will provide monthly status report to the HPEP Administrator/Manager and their respective management. The Commercial Laboratory PE Coordinator will interface with Quality Assurance staff and the QA/QC Officers at each of the Commercial Laboratories.

2.5 Laboratory QA/QC Officers

The participating laboratory QA/QC Officers will oversee the PE samples at each of the respective contract and commercial HPEP laboratories. The QA/QC Officers will coordinate the all sample tracking activities including receipt and documentation associated with any PE program material. The QA/QC officer will provide the PE Program Data Management Coordinator with historical performance data and current PE results as they are received by the laboratory.

The Laboratory QA/QC Officer will initiate the corrective action request, should the laboratory receive unsatisfactory results on any PE corrective action required as per program protocol. The QA/QC Officers will interface with the Special Project PE Coordinators to ensure that testing requirements are met.

3.0 PE PROGRAM - LABORATORY REQUIREMENTS

3.1 General Laboratory Requirements

Analytical laboratories providing services to the Hanford Site shall be able to demonstrate the technical competence in analyzing the samples they receive. This can be done by a number of methods, not limited to the list below.

- Matrix standards
- Round Robin with other laboratories
- Sample Splits with other laboratories
- Performance Evaluations (PEs)

3.2 Specific Laboratory Requirements

Each Laboratory shall be responsible for compliance with national regulator standards as applied to the analysis being performed, such as the Water Pollution (WP) exchange samples from EPA for laboratories analyzing water samples. Each laboratory participating in the project specific PE programs will receive a Letter of Instruction (LOI) outlining the analytical testing and reporting requirement.

3.3 Classification Of Samples

There are 3 classifications of samples that must be analyzed at the Hanford Site. High radiation (greater than 5 milli rem per hour on contact); Environmental level or low radiation (less than 5 milli rem per hour on contact) and non-radiation. Laboratories analyzing these class of samples must be specially equipped to analyze and dispose of these samples safely and to protect the environment and the public.

Appropriate requirements shall be employed at all times to ensure that safety, the environment, and the public are protected. These protection standards shall be ready for audit from local, federal, and state agencies.

3.4 PE Deliverable Requirements

Laboratories participating in Performance Evaluation programs are required to report in a specific format consistent with national standards, special project PE Program deliverables, or according to WHC contract requirements.

3.5 Communication of PE Results to the HPEP Data Management Coordinator

Contract Laboratories analyzing Hanford Site samples and participating in performance evaluations shall promptly report all performance information (including corrective actions) to the HPEP Data Management Coordinator. The Commercial Laboratory Coordinator will provide the inputs from Commercial Laboratories. This can be done by placing the HPEP Data Management Coordinator on distribution of all reports.

Early notification would be appropriate if the laboratory had failed to meet the PE standard.

3.6 Performance Evaluation Deficiency

When a participating laboratory fails acceptable performance on a PE and their recovery is not within control limits, the laboratory must investigate the non-conforming measurement system and identify the cause. Corrective action must be implemented and if possible another PE analyzed (if commercially available). All steps must be formally documented with a copy being sent to the HPEP Data Management coordinator.

3.7 Supplier Deficiencies

The laboratory or designated interface must notify the HPEP Administrator/Manager immediately and send a copy to the HPEP QA Officer when deficiencies or discrepancies are noted, upon receipt of HPEP originated PE material. The laboratory must notify the PE originator of deficiencies or discrepancies noted upon receipt of PE materials from other PE suppliers (i.e., EPA, DOE).

4.0 PERFORMANCE EVALUATION TOOLS

Performance evaluation tools are used to evaluate the performance of analytical support laboratories and the frequency of their use. These tools include laboratory audits, inspection of data deliverables, submission of PE materials blind to the laboratories, use of site-specific laboratory control samples (LCSs) and existing PE Program data. These tools are selected to provide information regarding the laboratories management practices as well as their performance in specific analytical disciplines.

For the purposes of this discussion, PE tools are referred to as "periodic" or "real-time", depending on the nature of their use. Periodic tools are used at a set frequency to provide information on laboratory performance, either general or discipline-specific, which is not specifically associated with a particular batch of Hanford Site field samples. Information supplied by periodic PE tools is indicative of overall performance or capabilities in a particular analytical discipline or method. Information provided by real-time tools are those which are either blind PE samples, laboratory QC, or assessment of a laboratories data deliverable.

4.1 General Laboratory Performance Evaluation Tools

Three performance tools are used to evaluate general laboratory operations: on-site audits, desk evaluations, and assessment of deliverables.

4.1.1 On-site Laboratory Assessments

Laboratory assessments are used as periodic tools to assess a laboratories capability to perform in accordance with contract and subcontract requirements. As a prerequisite to participation in the HPEP, each analytical

laboratory must pass an initial assessment to become OQA approved. Thereafter, each contract analytical laboratory is assessed on an annual basis, Commercial Laboratories on an 18 month basis or according to specific program requirements. These assessments may be performed by OQA personnel or other technical personnel from other Westinghouse Hanford Company (WHC) or DOE facilities. The assessments cover general laboratory operation, plus all analytical disciplines supported by the laboratory. As a condition of contract, all analytical laboratories must respond to findings incurred during an on-site assessment.

4.1.2 Desk Evaluations

Desk evaluations may be used in place of the 18 month on-site laboratory assessment. A desk evaluation includes review of the laboratory QA plan/program document, past assessment reports, any revised laboratory procedures, organizational and personnel qualifications, user references, performance and product history, state/industry certifications, and internal QA/QC practices.

If major problems are identified during a desk evaluation, an on-site assessment may be required.

4.1.3 Assessment of Data Deliverables

Assessment of analytical laboratory data deliverables against the Waste Analysis Plan (WAP), Statement of Work (SOW), Tank Waste Analysis Plan (TWAP), Test Plans, or Sample Analysis Plan requirements are used as a real-time assessment tool for general analytical laboratory operations. The data deliverable is evaluated for compliance with holding times, completeness and accuracy, as well as adherence to other specified requirements.

4.2 Method-Specific Laboratory Performance

Three assessment tools are used to evaluate laboratory performance within specific analytical disciplines or particular analytical methods. These are as follows:

- ~ Participation in existing PE programs
- ~ Analysis of HPEP specific PE samples
- ~ Routine laboratory QC samples.

4.2.1 Participation in Existing PE Programs

Results of participation in existing programs within the environmental analysis industry are used as periodic performance tools to check laboratory capabilities in specific analytical disciplines or methods. As a condition of contract, analytical laboratories are required to participate in certain programs for all sample matrices and analytical disciplines or methods for which they provide support to the Hanford Site, if these PE's exist. The requirements for participation come from several sources, (e.g., EPA CLP QB for CERCLA support); state certification (e.g., drinking water analyses); DOE mandates (e.g., Orders, QAP and RIS), or the HPEP requirements. Costs associated with participation in these programs shall be absorbed by the laboratories as a cost of doing business with the Hanford Site. As a

condition of HPEP participation, analytical laboratories must agree to make the results in these programs available to the HPEP Administrator/Manager or Special Laboratory PE Coordinators.

Materials used in existing programs are formulated by those programs to meet their own needs. While these materials are extensively characterized, the matrix, choice of analytes, and concentration levels may not match those of the Hanford Site field samples. Thus, the ability to successfully analyze these materials may not reflect the ability to successfully analyze Hanford Site field samples (especially tank waste). Therefore, results from participation in these programs are used by the HPEP to assess the general ability of a laboratory to perform on a generic matrix.

4.2.2 Existing PE Programs

1. *Environmental Monitoring Systems Laboratory, Las Vegas (EMSL-LV) Radiological Intercomparison Study (RIS)*: All laboratories providing environmental level radiochemical services (gross radiological screening as well as specific radionuclide analyses) shall participate. Available matrices are water and air filters.
2. *Environmental Measurements Laboratory (EML) Quality Assessment Program (QAP)*: All laboratories providing environmental level radiochemical services must participate semiannually in the program. Available matrices are water, soil, air filters, and biota (vegetation).
3. *EPA CLP Quarterly Blind (QB) Performance Evaluation*: All analytical laboratories supporting metals, VOC, SVOC, pesticide/PCB or cyanide analyses via CLP methods must participate in the QB Program for those analytical disciplines and matrices applicable to the support provided by the laboratory. Laboratories supporting SDWA analyses are exempt only from required CLP QB participation. Those analytical laboratories that are participants in the CLP must participate each quarter as part of their CLP contract. Those laboratories that are not members of the CLP must participate in the QB program at a minimum of semiannually as long as an agreement exists between EPA and DOE allowing such participation. The PE program manager is responsible for providing QB samples to non-CLP laboratories. An example of a non-CLP laboratory on the Hanford Site would be the 222-S Laboratory.
4. *Water Pollution (WP) Performance Evaluation Program*: All laboratories that analyze Hanford Site water, soil, or waste samples using CWA or RCRA methods must participate in the semiannual WP program sponsored by Environmental Monitoring Systems Laboratory, Cincinnati (EMSL-CI) for all analytes and methods required by HPEP. (WP participation for RCRA support is a HPEP imposed requirement in order to provide some performance data on the determinative steps of RCRA analyses.)
5. *Water Supply (WS) Performance Evaluation Program*: All laboratories that analyze Hanford Site samples using SDWA methods must participate in the EMSL-CI's semiannual WS program for all analytes and methods required by HPEP. Laboratories analyzing SWDA samples

must maintain current Washington State certification for required analytes.

6. *Radiological and Environmental Sciences Laboratory (RESL) Mixed Analyte Performance Evaluation Program (MAPEP)*: Participation in this semiannual DOE-sponsored program, which is still under development, will be required for all laboratories supporting environmental level radiochemical and metals analyses of radiologically contaminated water and soil on the Hanford Site.
7. *American Industrial Hygiene Association*: This national program maintains three proficiency programs, two of which are associated with laboratory accreditations. Laboratories performing related analyses must remain proficient in these programs to maintain the related accreditations.
  - The Proficiency Analytical Testing (PAT) Program provides quarterly samples for accreditation of general industrial hygiene analyses. Each sample set includes four filter or sorbent tube samples and a blank in the areas of fiber counting for asbestos analysis, metals analysis (3 metals/filter), organic solvent analysis (1-3 solvents/sorbent tube), and free silica analysis.
  - The Environmental Lead Proficiency Analytical Testing (ELPAT) Program provides quarterly samples for accreditation of laboratories performing analysis of lead in paints, swipes, or soils. Four samples of each matrix, plus a blank for swipes, are provided. Currently only paint and swipe samples and accreditations are pertinent to Hanford laboratory work.
  - AIHA also sponsors a non-accreditation proficiency program for bulk asbestos identification using polarized light microscopy. Four bulk samples per quarter are provided in this program.
8. *United States Council on Energy Awareness (USCEA) - National Institute of Standards and Technology (NIST) Radioactivity Measurements Assurance Program*: This program operates approximately every two months, with the results due approximately three months later. This program tests radioisotopes at concentrations and matrix most consistent with the nuclear power industry. Reports of a laboratory's traceability to NIST are issued.

5.0 PERFORMANCE ASSESSMENT (AUDITS, PERFORMANCE EVALUATIONS AND INTERNAL QA)

5.1 Audits, Performance Evaluations and Internal QA

There are 3 levels of effort that will help in providing assurance that the laboratory is maintaining its ability to analyze Hanford Site samples.

First, regularly scheduled technical and compliance audits will provide basic assurance that the laboratory exercises "good laboratory practices" and that analytical results are valid.

Second, Quality Assurance, internal to HPEP, would provide evidence that the laboratory, in question, is providing valid data consistently. Various methods would include, but would not be limited to:

- Providing justifications for the purchase of commercially available blind standards that the laboratory would be expected to analyze.
- Splitting a Hanford Site sample between 2 or more laboratories. (Protocols would have to be established to provide evidence of sample integrity.)
- Splitting a Hanford Site sample and sending it to the laboratory as a "double blind".
- Arranging for all involved laboratories to analyze commonly agreed upon material as a "Round Robin".
- Developing a material that can be sent randomly and covertly to the laboratory that would be masked as a Hanford Site sample.

Third, performance evaluations from independent sponsors would provide further assurance of valid data. They also provide a reference of how the laboratory performs in comparison with other laboratories performing the same work. Care must be taken so that the samples analyzed can be related to the Hanford Site samples that the laboratory is commissioned to evaluate.

Section 8.0 lists the various laboratories involved with Hanford Site sample analysis and the exchange programs that they are involved with.

5.2 Tracking and Trending

Laboratories, analyzing Hanford Site samples, shall maintain a data base of their performance evaluations. This data should be used to evaluate trends (if any) developing within each of their performance programs.

The HPEP Data Management Coordinator shall keep data bases of all laboratories analyzing Hanford Site samples and will include current information in the monthly HPEP data management status report (Section 7.2.2.2).

The purpose of the performance indicator is to identify, as early as possible, trends that may eventually lead to reporting of data of unknown quality.

6.0 PERFORMANCE STATUS

The HPEP will status participating laboratories, based upon their performance in their respective programs and the applicability of the analysis being performed to the matrix and concentration of the exchange sample. The performance statuses are:

- Satisfactory - The laboratory is participating in an established program and the performance has generally been within acceptable limits.
- Unsatisfactory - The laboratory is participating in an established program, and the performance has not been within acceptable limits frequently.
- Trial - The laboratory has entered into new types of analyses and would like to participate in the program to see if qualification can be established.
- Inactive - The laboratory had participated in a program; performance results were either unsatisfactory and had stopped analyzing samples related to the exchange; the exchange was not related to the types of samples analyzed.

6.1 Performance Status Change

Status may be changed for the following reasons:

- The laboratory does not analyze Hanford Site samples in the concentration or matrix of the exchange. This may be over ridden if other certification requirements are to the contrary.
- The laboratory no longer analyzed samples related to the exchange.
- Laboratory performance on the exchange is unsatisfactory and cannot regain qualification; and is not permitted to report results to the samples.

6.2 Addition of a PE Program

Addition of a PE program would be a small part of a large program to establish new work or technology needed for the cleanup mission. HPEP shall assist in establishing enrollment and provide advice necessary for startup. Documentation and implementation will be coordinated with the Special Laboratory and the Project Representative.

6.3 Termination of a PE Program

HPEP will assist with terminating PE programs. Care must be taken to make sure that the cause for termination is justified and that the termination will not cause unexpected impacts, on the laboratory and/or the Hanford Site Program. All participating parties in the PE program must agree on the justifications for elimination (including regulatory approval).

6.4 Notification of Performance

It is the responsibility of HPEP to notify participating laboratories in writing, if the status of the laboratory changes.

7.0 QUALITY ASSURANCE REQUIREMENTS

The HPEP is an important component in the effort to assure the accuracy of analytical laboratories performing analyses for the Hanford Site. The data (results) from a laboratory's participation in the HPEP, coupled with the performance information provided by the other programs the laboratory, is the measurement by which the laboratory will be assessed. Performance evaluations are commonly called performance audits in laboratory quality assurance plans or programs.

7.1 Nonconformance and Corrective Action

Programmatic nonconformances, corrective actions, and conditions adverse to quality occurring within an HPEP laboratory shall be documented and resolved as per the requirements of that laboratory's Quality Assurance Plan. Typically, unacceptable performance evaluation results shall require corrective action, as per program protocol. Corrective action documentation shall be forwarded to the PE Program Data Management Coordinator. This requirement will be fulfilled by communication of the results and corrective actions coordinated by the Laboratory QA Officer. This information will be forwarded within a reasonable time after the receipt of the results by the laboratory.

The HPEP Administrator/Manager is responsible for ensuring that all nonconformances, corrective actions, and conditions adverse to quality are resolved in a timely manner.

7.2 Quality Assurance Reports to Management

7.2.1 Reports to the OQA Manager (HPEP Administrator/Manager) will be prepared by the HPEP Data Management Coordinator and HPEP QA Officer.

A quarterly PE status report will be submitted to the Analytical Services Manager and DOE/RL. This report shall address any changes in a laboratory's performance status and any outstanding PE related nonconformances or corrective actions. Copies of the quarterly report will be sent to the laboratory manager and laboratory QA officer; as appropriate.

7.2.2 Reports to the PE Program Manager

7.2.2.1 A monthly laboratory PE status report will be submitted to the HPEP Administrator/Manager. This report will be prepared by the Laboratory QA Officer and the special Project PE Coordinator. This can simply be a routed copy of the monthly QA status report that is part of a requirement presently driven by each Laboratory's QA Plan/Program. This report can include a Performance Evaluation or Performance Audit Section, or a stand-alone report.

7.2.2.2 A monthly PE data management status report will be submitted to the HPEP Administrator/Manager and will be prepared by the PE Program Data Management Coordinator. This monthly summary report will include status of all laboratories performing analyses for the Hanford Site. This report will contain performance indicators and performance status for each laboratory.

8.0 LABORATORY PE PROGRAM INFORMATION

8.1 Laboratory PE Participation

8.1.1 222-S Laboratory

222-S Laboratory Analytical Operations manages the 222-S Laboratory facilities in performing radioanalytical, inorganic, and organic chemistry analyses in support of WHC environmental programs and waste management activities. A program support group provides project coordination to enhance laboratory quality and throughput. Also provided are Hazardous Material Control activities, to ensure proper chemical storage; Building Operations, to assure facilities are maintained and to manage maintenance and construction activities. Shift Operations provides around-the-clock analytical capability. Refer to Table 1 for 222-S Laboratory PE program participation.

Table 1. 222-S Laboratory Classification - High Level Radiation.

CERTIFICATION REQUIRED	CERTIFIED	PE NAME	TYPE OF PE PROGRAM	DRIVER
Yes*		EMSL-RIS	Environmental Level Radiological	WAC 173-50
N/A		EML-QAP	Environmental Level Radiological	DOE
N/A		QB	EPA (CLP- Water & Soil)	
Yes		WP	Waste Water	WAC 173-50
N/A		MAPEP**	Environmental Level Radioactive Mixed Waste	DOE/HQ
N/A		SEE	Waste Tank matrices - Pilot Program (Referee)	TWRS
N/A		LIBS	Laboratory Initiated Blind Standards	Internal QA

* Laboratories classified as High Radiation Laboratories are exempt and are not required to be certified by the Washington State. They may participate to demonstrate flexibility and proficiency. Presently there are no programs available to certify laboratories for high level radiological analyses. Refer to Section 9.1, the SEE Program for more information.

** Laboratories classified as High Radiation Laboratories are exempt and not required to participate. Laboratories may participate to demonstrate flexibility and proficiency.

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8.1.2 Waste Sampling and Characterization Facility Laboratory (WSCF)

WSCF Analytical Operations manages the WSCF complex and provides analytical laboratory services which include:

- Radiological counting room support in stack and room air monitoring and low-level environmental
- Process control support to liquid effluent treatment systems
- Analysis of samples for safe drinking water
- Support analyses of breathing zone
- Bulk samples for the presence of asbestos fibers
- Support analysis of Industrial Hygiene sampling at the work place.

Refer to Table 2 for WSCF Laboratory PE program participation.

Table 2. WSCF Laboratory Classification - Low (Environmental) Level Radiation.

CERTIFICATION REQUIRED	CERTIFIED	PE NAME	TYPE of PE PROGRAM	DRIVER
Yes	Yes	EMSL-RIS/EPA	Environmental Level Radiological	WAC 173-50
N/A		EML-QAP/DOE	Effluent Monitoring	DOE
N/R		QB/EPA	Contract Laboratory Program (Water & Soil)	
Yes	Yes	WP	Waste Water	WAC 173-50
Yes	Yes	WS/WSQB*	Drinking Water	WAC 173-50
Yes	Yes	Asbestos Fiber Counting Filter (PAT*)	Asbestos	AIHA
Yes	Yes	Bulk Asbestos	Asbestos	AIHA
Yes	Yes	Metals- Air Filter	Metals	AIHA
Yes	Yes	Organics - Sorbet Tubes	Organics	AIHA
Yes	Yes	Free Silica - Filter	Silica	AIHA
Yes	Yes	Bulk Asbestos	Asbestos	NIST - NVLAP
Yes	Yes	Environmental Lead Paint (ELPAT**)	Lead	AIHA
Yes	Yes	ERA/APG	Accreditation	WDOE
Yes	No	Round Robin	Asbestos	AIHA

* = WSQB is part of the WS Program, required to analyze quarterly blind samples

8.1.3 PNL Laboratories

PNL Laboratories PE Programs will be incorporated in HPEP Revision 1. Refer to Table 3 for PNL Laboratories PE Program Participation.

Table 3. PNL Laboratories

CERTIFICATION REQUIRED	CERTIFIED	PE NAME	TYPE of PE PROGRAM	DRIVER
Yes*		EMSL-RIS	Environmental Level Radiological	WAC 173-50
N/A		EML-QAP	Environmental Level Radiological	
N/A		QB	EPA (CLP- Water & Soil)	
Yes		WP	Waste Water	WAC 173-50
N/A		MAPEP**	Environmental Level Radioactive Mixed Waste	DOE/HQ
N/A		NIST	Radiological	Internal QA
N/A		LIBS	Laboratory Initiated Blind Standards	Internal QA

* Laboratories classified as High Radiation Laboratories are exempt and are not required to be certified by the Washington State. They may participate to demonstrate flexibility and proficiency. Presently there are no programs available to certify laboratories for high level radiological analyses. Refer to Section 9.1, the SEE Program for more information.

** Laboratories classified as High Radiation Laboratories are exempt and not required to participate. Laboratories may participate to demonstrate flexibility and proficiency.

8.1.4 WHC Commercial Laboratories

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8.1.4 WHC Commercial Laboratories

LABORATORY	CERTIFICATION REQUIRED	CERTIFIED	PE NAME	TYPE OF PE PROGRAM	DRIVER
QES/KNOXVILLE			WS WP	Water Studies Waste Water	WAC 173-50 WAC 173-50
QES/ST. LOUIS			WS WP EMSL EML MAPEP	Water Studies Waste Water Environmental Radiological Level Environmental Radiological Level Environmental Level Radioactive Mixed Waste	WAC 173-50 WAC 173-50 DOE
QES/RICHLAND			EMSL	Environmental Radiological Level	
LOCKHEED			WP WS EMSL EML MAPEP	Waste Water Water Studies Environmental Radiological Level Environmental Radiological Level Environmental Level Radioactive Mixed Waste	WAC 173-50 WAC 173-50 DOE

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8.2 Laboratory PE Distribution Schedule

Distribution schedules are only for Hanford Site Laboratories at this time. Commercial Laboratory contracts will be added at a future date.

8.2.1 222-S Laboratory

Refer to Table 4 for 222-S Laboratory PE distribution schedule.

Table 4. 222-S Laboratory 1995 PE Distribution Schedule.

JANUARY	FEBRUARY	MARCH	APRIL	MAY	JUNE
EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Gross A, B Sr 89-90 DOE-RESL Mixed Waste (MAPEP) Inorg/Rad Liquid	EPA-RIS (EMSL) U-Radium	EPA-WP (Inorg/Org) EPA-RIS (EMSL) H-3, U, Pu Air Filter DOE-EML QAP (Rad) SEE (Inorg/Org/Rad)	EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Performance Eval. A & B (Rad)		EPA-RIS (EMSL) Mixed Gamma U-Radium DOE-RESL Mixed Waste (MAPEP) Inorg/Rad Soil/Sed.
JULY	AUGUST	SEPTEMBER	OCTOBER	NOVEMBER	DECEMBER
EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Gross A, B Sr 89-90	EPA-RIS (EMSL) Uranium	EPA-WP (Inorg/Org) DOE-EML QAP (Rad) EPA-RIS (EMSL) Air Filter SEE (Inorg/Org/Rad)	EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Performance Eval. A & B (Rad)	EPA-RIS (EMSL) Alpha-Beta H-3 Mixed Gamma	

Note: This schedule does not include Laboratory QA initiated Blinds (e.g., ERA or APG). Laboratory QA initiated blinds are issued as necessary (ad hoc). Therefore frequency and times will vary. EMSL distribution may vary.

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8.2.2 WSCF Laboratory

Refer to Table 5 for WSCF Laboratory PE distribution schedule.

Table 5. WSCF Laboratory 1995 PE Distribution Schedule.

JANUARY	FEBRUARY	MARCH	APRIL	MAY	JUNE
EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Gross A, B Sr 89-90 AIHA-PAT	EPA-RIS (EMSL) U-Radium ELPAT-Bulk	EPA-WP (Inorg/Org) EPA-RIS (EMSL) H-3, U, Pu Air Filter DOE-EML QAP (Rad) WSQB	EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Performance Eval. A & B (Rad) AIHA-PAT NVLAP	ELPAT-Bulk	EPA-RIS (EMSL) Mixed Gamma U-Radium WA-WS (Inorg/org) WSQB
JULY	AUGUST	SEPTEMBER	OCTOBER	NOVEMBER	DECEMBER
EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Gross A, B Sr 89-90 AIHA-PAT	EPA-RIS (EMSL) Uranium ELPAT-Bulk	EPA-WP (Inorg/Org) WSQB DOE-EML QAP (Rad) EPA-RIS (EMSL) Air Filter	EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Performance Eval. A & B (Rad) WA-WS (Inorg/Org) AIHA-PAT	EPA-RIS (EMSL) Alpha-Beta H-3 Mixed Gamma ELPAT-Bulk NVLAP	WSQB

Note: This schedule does not include Laboratory QA initiated Blinds (e.g., ERA or APG). AIHA and Laboratory initiated blinds are issued as necessary (ad hoc). Therefore frequency and times may vary. EMSL distribution may vary.

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8.2.3 PNL Laboratories

Refer to Table 6 for PNL Laboratories PE distribution schedule.

Table 6. PNL Laboratories 1995 PE Distribution Schedule.

JANUARY	FEBRUARY	MARCH	APRIL	MAY	JUNE
EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Gross A, B Sr 89-90 DOE-RESL Mixed Waste (MAPEP) Inorg/Rad Liquid	EPA-RIS (EMSL) U-Radium NIST	EPA-WP (Inorg/Org) EPA-RIS (EMSL) H-3, U, Pu Air Filter DOE-EML QAP (Rad) SEE (Inorg/Org/Rad)	EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Performance Eval. A & B (Rad) NIST		EPA-RIS (EMSL) Mixed Gamma U-Radium DOE-RESL Mixed Waste (MAPEP) Inorg/Rad Soil/Sed. NIST
JULY	AUGUST	SEPTEMBER	OCTOBER	NOVEMBER	DECEMBER
EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Gross A, B Sr 89-90	EPA-RIS (EMSL) Uranium NIST	EPA-WP (Inorg/Org) DOE-EML QAP (Rad) EPA-RIS (EMSL) Air Filter SEE (Inorg/Org/Rad)	EPA-CLP (Inorg/Org) EPA-RIS (EMSL) Performance Eval. A & B (Rad) NIST	EPA-RIS (EMSL) Alpha-Beta H-3 Mixed Gamma	

Note: This schedule does not include Laboratory QA initiated Blinds (e.g., ERA or APG). Laboratory QA initiated blinds are issued as necessary (ad hoc). Therefore frequency and times will vary. EMSL and NIST distribution may vary.

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9.0 PROJECT SPECIFIC PE PROGRAMS (REFEREE/SPLIT STUDIES)

9.1 Sample Exchange/Evaluation Program (Referee Program)

The SEE Program is a joint effort of WHC and Pacific Northwest Laboratories (PNL) to compare analytical laboratory performance samples from the Hanford Site's high level radiation waste tanks. Phase I of the SEE Program compared radiochemical results on samples from two waste tanks, B-111 and T-107. In Phase II the program was expanded to include inorganic constituents in addition to radionuclides.

Phase I and II sample exchange results have shown that the analytical methods tested from WHC and PNL produce comparable results on homogeneous waste solutions. Results from Phase I and II have provided sufficient information to permit the SEE program to initiate testing of procedures on solid waste samples. Phase III will include testing of laboratory digestion procedures and analytical methods that are normally applied specifically to high level radiological solid waste sample analyses. See Appendix A for a status report on the Sample Exchange/Evaluation Program Phase III.

9.2 Split Sample Study

A concern of quality control in performing chemical sampling and analysis is the comparability of analytical data between one laboratory and another. One method of evaluating this comparability is the review of data from split samples. Split samples are taken from as homogeneous a sample as possible. They are considered identical. These can be in any number, but are done here only in pairs. Each split from the main sample is sent to a different laboratory. The master sample is sent to the primary laboratory and its identical sample or "split sample" is sent to an alternate laboratory. The data from these matched master and split samples are compared through data assessment.

The tests allow a focus on potential differences in laboratory analyses. If the user observes a statistical difference that is near an action level the lack of comparability should impact decisions made on the results. Conversely, the user may observe a statistical difference between laboratories and yet the ranges of data from both labs may be all below (or all above) an action level. The lack of comparability in this latter situation need not impact decisions based on the data. Nonetheless, any decisions made with biased data may have to account for the magnitude of the bias.

See Appendix B for the results of the Comparison of Master and Split Sample Results.

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10.0 REFERENCES

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APPENDIX A

SAMPLE EXCHANGE/EVALUATION (SEE) REPORT PHASE III

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APPENDIX A**SEE Program Phase III - Test Plan and Guidance Instructions****1. Introduction**

This test plan describes the implementation of the third phase of the Sample Exchange/Evaluation (SEE) Program. This Test Plan is divided into the following sections:

- 01 Introduction
- 02 Objectives
- 03 Schedule
- 04 Data Reporting/Review
- 05 Final Report Preparation

The SEE Program is managed by a collaborative called the QA/QC Triad (Triad). The Triad is made up of members from the PNL Analytical Chemistry Laboratory (ACL), the WHC Analytical Services 222-S Laboratory, and the WHC Analytical Services (AS) Office of Quality Assessment. The analytical laboratories participating in this Phase of the program will be the PNL ACL and WHC AS 222-S.

2. Objectives

The primary objective of Phase III is to provide comparison data for the laboratory sample preparation methods (i.e., fusion, acid digest, water leach) and analysis of the sample as received (direct). This objective will be accomplished by providing each participating laboratory with an aliquot of an homogenized core sample. The ACL will provide the core material to be used in this Phase of the program.

The ACL will homogenize and distribute the core material to the PNL ACL and WHC AS 222-S laboratories. Upon receipt of the homogenized core material at each laboratory, the sample material will be re-homogenized. Sets of four aliquots will be prepared for analysis by fusion, acid digestion, and water leach. An additional four aliquots will be analyzed directly for mercury by Cold Vapor Atomic Absorption (CVAA), total organic carbon/total inorganic carbon/total carbon (TOC/TIC/TC), cyanide, and differential scanning calorimetry/thermogravimetric analysis (DSC/TGA). Each sample will be prepared/analyzed to provide a measure of the precision of each laboratory's homogenization and preparation techniques.

Some measurement methods from previous program phases where additional data is desired will also be investigated. These include fluoride ion (an evaluation of Ion Chromatography (IC) versus Ion Selective Electrode (ISE)), total alpha, and Inductively Coupled Plasma (ICP) metals determinations. The prepared sample solutions will be analyzed in accordance with the matrix shown in Table 2.0 and the attached flow diagram.

In addition to the evaluation of sample preparation methods, single blind QC standards will also be submitted in Phase III to provide both performance evaluation and benchmark information on analytical methods employed under this study. The QC standards employed in this Phase will be prepared by the WHC 222-S Standards Laboratory. Each blind standard will be analyzed in duplicate.

Table 2.0 Sample Analysis Matrix

Objective 1 - Sample Prep Method Comparison			
Direct	Water Leach	Acid Digestion	Fusion
CVAA (Hg) TOC/TIC/TC Cyanide DSC/TGA	IC - Anions (F, Cl, NO ₂ , NO ₃ , SO ₄ , PO ₄) F- by ISE TOC/TIC/TC (QC Standard Only)	ICP - Metals *	Gamma Energy Analysis (GEA) - all detected isotopes Total Alpha Total Beta Pu-239/240 SrY-90 Tc-99 * Uranium ICP-Metals *

* Specific analytes will be determined when tank core material is obtained and will be documented in the shipping papers and any test instructions accompanying the samples.

3. Schedule

The ACL will prepare the homogenized core material within 45 days of issue of the WHC Letter of Instruction, with funding, requesting initiation of work. The samples will be shipped to participating laboratories within the 45 day time line identified above. Analytical data will be submitted to the WHC Office of Quality Assessment within 60 days of receipt of the homogenized core material by the laboratories. A final report will be submitted to Tank Waste Remediation Systems (TWRS) and laboratory management 60 days following receipt of all data by the WHC Office of Quality Assessment.

4. Data Reporting/Review

Data from the ACL will be reported to the ACL QO&SL Manager. Data from 222-S will be reported to AS Program Support. Each laboratory will tabulate and submit the analytical results to the WHC Office of Quality Assessment. All radiochemistry data will be reported in units of $\mu\text{Ci/g}$ (in the homogenized core material, as received). All radiochemical results shall include uncertainty, the basis for the uncertainty reported, and the minimum detectable activity (MDA) for each method. All other data will be reported in units of $\mu\text{g/g}$ (in the homogenized core material, as received). All inorganic results shall include the method detection limit (MDL) for each analyte reported. Data for the blind QC Standard will be reported in $\mu\text{Ci/ml}$ or $\mu\text{g/ml}$ based on the as-received solution. The WHC Office of Quality Assessment will conduct a point-by-point comparison, calculate mean relative percent difference (mRPD) for both the standards and preparation sample results, calculate the accuracy for all the standard results, and tabulate the data. Additional statistical analysis of the data will be performed by the 222-S statistical analysis group. The results will then be evaluated using the following criteria as a guide:

The following evaluation criteria will be used only when the reported results have counting errors $< \pm 15\%$ (1 sigma) for radiochemical results or are 5 times the MDL for inorganic results.

1. If mRPD < 20% no action.
2. If mRPD > 20% < 50% prepare corrective action with cognizant scientist(s).
3. If mRPD > 50% immediately notify lab management (AG King, ACL; ML Bell, AS), prepare corrective action plan with cognizant scientist(s).

Radiochemistry values which have counting errors > $\pm 15\%$ (1 sigma) and are above the MDA will be investigated if the reported results do not overlap at the 3 sigma confidence level. Inorganic values which are above the MDL, but less than 5 times the MDL shall be investigated if the reported results differ by more than 2 times the MDL.

Corrective action(s), as appropriate, will be prepared by the WHC Office of Quality Assessment based on input from the ACL and WHC 222-S representatives.

5. Final Report Preparation

A final report will be prepared and delivered to the Test Plan Approval Authorities within 60 days of receipt of all data generated under this Phase of the program by the WHC Office of Quality Assessment.

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APPENDIX B

SPLIT SAMPLE COMPARABILITY STUDY

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APPENDIX B

Split Sample Comparability Study

Comparison of Master and Split Sample Results

Date Published
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EXECUTIVE SUMMARY

CERCLA Ground Water Monitoring data were evaluated for comparability. When a master sample's analyzed value and its respective split sample's analyzed value are statistically indistinguishable, the analyses are considered comparable. In other words, the master and split sample values come from the same population and either one represents the same result. If they are not comparable, there is a difference between the laboratory producing the master sample value and the laboratory producing the split sample value.

The Paired-t Test and the Wilcoxon Signed Ranks Test were applied to data. For 3 of 7 analytes evaluated, the data were comparable at the 95% confidence level. The remaining four were not comparable.

The tests allow a focus on potential differences in laboratory analyses. If the user observes a statistical difference that is near an action level the lack of comparability should impact decisions made on the results. Conversely, the user may observe a statistical difference between laboratories and yet the ranges of data from both labs may be all below (or all above) an action level. The lack of comparability in this latter situation need not impact decisions based on the data. Nonetheless, any decisions made with biased data may have to account for the magnitude of the bias.

It is recommended the procedure be applied to evaluate comparability. First, it is the only test in hand to evaluate this quality parameter, as required by several Project Plans. Second, this has proven to be a workable means of testing laboratory data comparability. This procedure had been adapted to and tested on the data of concern - environmental analyses at the Hanford Site. Consequently, it is relatively simple to use on data extracted from the same database. The level of rigor is appropriate and effective for evaluating split sample data.

ACKNOWLEDGEMENT

The authors acknowledge the contribution of Charissa Chou who recommended the statistical tests used, provided background on statistical methods, and provided prototype spreadsheets and templates to illustrate methodology. She also gave guidance on how to treat statistical outliers in the data.

The authors acknowledge the support provided by Sean Reffalt and Roger Price in supplying data and providing the needed knowledge of the contents and format of the HEIS database.

1.0 INTRODUCTION

A concern of quality control in performing chemical sampling and analysis is the comparability of analytical data between one laboratory and another. One method of evaluating this comparability is the review of data from split samples. Split samples are taken from as homogeneous a sample as possible. They are considered identical. These can be in any number but are done only in pairs here. Each split from the main sample is sent to a different laboratory. The master sample is sent to the primary laboratory and its identical sample or "split sample" is sent to an alternate laboratory. The data from these matched master and split samples are compared through data assessment.

The authors tailored an existing method for evaluation of split sample results. In developing the method, an initial evaluation of comparability was made as well.

2.0 OBJECTIVES

The evaluation objectives were threefold, to:

1. Develop a process for comparing master and split sample results
2. Test the process on a collection of real data
3. Evaluate the process and its potential to assure quality data.

These objectives were accomplished.

3.0 DISCUSSION

The HEIS database is a convenient source for data, particularly considering the capability for obtaining the data. HEIS personnel extracted CERCLA Project Ground Water data for seven projects from the HEIS database. This Ground Water data extended over several years. Data was selected using following guidelines:

- Data flagged "R" or "U" were deleted.
- Statistical outliers, determined by the Box and Whisker test, are eliminated from the statistical analyses of the data. (Review of the data used herein showed the final decision remained the same whether or not outliers were included.)
- Seven common analytes were to be evaluated.

Data used in statistical tests were the differences between each master and split sample pair. The null hypothesis is that analytical results produced by the two laboratories are the same. Two tests were used; the Paired t-test and the Wilcoxon Signed Ranks Test. The significance value, alpha, was set at 0.05. **Where the null hypothesis is not rejected, the conclusion is that the data are comparable.** If the null hypothesis is rejected, the data are not comparable.

The Paired t-test is applicable for large numbers of samples with normally distributed data. In contrast, the Wilcoxon Signed Ranks Test is applicable to either a small or large number of samples, even those with non-normal distributions. Both the Paired t-test and the Wilcoxon Signed Ranks Test were applied to the seven analytes' results. The normality of each data set was evaluated using the Lilliefors test at a 5% significance level.

A number of computer codes were employed in the evaluation. HEIS provided the data. The data was prepared in PARADOX to create useful formats for data review and for export to the other evaluation codes. STATGRAPHICS was used to determine if any data points were considered statistical outliers using The Box and Whisker test within STATGRAPHICS. *Although data was not eliminated from the conclusions below on this basis, the impact of statistical outliers was evaluated.* The data was exported to LOTUS for performing the Paired t-test and the Wilcoxon test. A combination of LOTUS functions and manual data manipulation accomplished these tests. Also, plots of the data afforded "common sense" graphical checks. QUATTRO PRO was used to produce precisely sized plots for use with a template. These plots permitted application of the Lilliefors test for data distribution normality. This normality test can be performed using the normality test within STATGRAPHICS eliminating the need for QUATTRO PRO software.

Conclusions were drawn based on the test results. The Paired t-test was not the test of choice where data was not normally distributed.

4.0 SUMMARY OF RESULTS

Table 1 presents a summary of results. The left column lists the seven analytes chosen. The next two columns, Lab A High and Low Values, give perspective to the range of sample values. Compare each range to the fourth column. This gives the difference between the Lab A value and the Lab B value. The fifth column gives the Lilliefors test result for data distribution normality. The data can be reasonably approximated by normal distributions in only two cases.

The final two columns give the statistical test results. The Paired t-test rejects the null hypothesis for only one, but the data for another five analytes do not meet the normal distribution criterion for using the Paired t-test. The Wilcoxon Test, which does not assume a normal distribution, must be the basis for conclusions on comparability for these five analytes. The Wilcoxon Test does "Not Reject" the null hypothesis for three of the seven analytes.

For normally distributed data, arsenic and fluoride, the Paired t-test and Wilcoxon test agree.

5.0 CONCLUSIONS

The data are generally not normally distributed. (This result was expected by those with experience with "real" data on the site.)

Four of seven analytes have values which are not comparable at the significance level used. The Wilcoxon Test is effective at making this distinction. The distinction is not easily made by "observing" a plot of Laboratory A versus Laboratory B values. A statistical procedure has proven to be a more reliable judge of comparability than visual appraisal of a plot.

An effective, appropriate procedure for evaluating split samples includes the following steps:

- Develop guidelines for accepting data, i.e., "R" and "U" data should be removed from further evaluation. Data flagged by other qualifiers by the laboratory or data validators are included in the statistical test.

- Determine whether sample data differences have normal or non-normal distributions.
- Determine outliers.
- Conclude reject/not reject and determine actions to be taken for reject results based on DQO requirements.

The impact of statistical outliers does not appear to be significant in this evaluation. Statistical outliers tend to improve the Paired t-test statistic, in the direction that the data are comparable. This is mitigated because data with outliers tend to be non-normal, favoring the power of the Wilcoxon Test over the Paired t-test.

6.0 RECOMMENDATIONS

For future application of statistical tests for comparability, the specific application chosen should result from considering factors such as project importance, the emphasis on specific analytes, any indications that laboratories have difficulty in analyzing certain (key) analytes, etc. The objectives of this procedure development favored the choice of common analytes, projects with significant numbers of split samples, and the simplicity of a water matrix.

The following strategy will best utilize this strategy:

1. Use the 7-step DQO process (see EPA 1993) to: (a) identify the key constituents of concern for a particular site; (b) determine the number of sample sizes needed for future comparability evaluation studies (rather than evaluate the data after data are collected); (c) determine when to do some follow-up actions (e.g., audit). When the 7-step DQO process is used, one knows what are the magnitudes of decision errors and also direct one's limited resources toward key "decisional" constituents of concern. Obviously one can tolerate larger magnitudes of differences for some constituents (because these constituents are not used in the "regulatory" decision making process) and at some concentration ranges (because these ranges are much higher than the regulatory "threshold").

2. Use PE samples in addition to field splits.
3. Examine the feasibility of streamlining the process by utilizing ready-made computer codes and/or developing a cookbook approach to facilitate the process of evaluation.

Table 1. Summary of Results

	Lab A High Value	Lab A Low Value	Normal Distri- bution(?)	Paired t-test Result	Wilcoxon Test Result
Arsenic	9.2	2	Yes	Reject	Reject
Calcium	111,000	8,080	No	Reject	Reject
Chromium	200	6	No	Not Reject	Not Reject
Fluoride	1.7	0.5	Yes	Not Reject	Not Reject
Iron	1030	10.3	No	Not Reject	Reject
Lead	10.5	2	No	Not Reject	Not Reject
Nitrate	80.5	0.9	No	Reject	Reject

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