PROCEDURES INCLUDED IN SAP

SAP/ESH Plan Call-out	New Procedure Number	Procedure Title
Procedures		
CDM-029	PRS-ENM-0026	*Wet Chemistry Data Verification and Validation*
EH-4516	PRS-RAD-1109	*Radioactive Contamination and Control Monitoring*
EH-4527	PRS-RAD-1105	*Receipt, Transport, and Movement of Radioactive Materials*
ENR-0020	PRS-ENR-0020	*Direct Push Technology Sampling* (as appropriate)
ENR-0021	PRS-ENR-0021	*Tree Sampling*
ES-1001	PRS-ENM-1001	*Transmitting Environmental Data to the Oak Ridge Environmental Information System OREIS*
ES-1002	PRS-ENM-1002	*Submitting, Reviewing, and Dispositioning Changes to the Environmental Information Management (EIM) Databases (OREIS, PEMS, and TRACKER)*
ES-1510	PRS-ENM-5004	*Subcontracting Analytical Work and Sample Material Tracking*
ES-2000	DELETED	*Field Screening Using an INNOV-X Systems Hand-Held X-Ray Fluorescence Spectrometer*
ES-2300	PRS-ENM-2300	*Collection of Soil Samples*
ES-2700	PRS-ENM-2700	*Field Logbooks*
ES-2702	PRS-ENM-2702	*Decontamination of Sampling Equipment and Devices*
ES-2704	PRS-ENM-2704	*Trip, Equipment, and Field Blank Preparation*
ES-2708	PRS-ENM-2708	*Chain of Custody Protocol for Environmental Sampling*
PA-5107	PRS-ENM-5107	*Inorganic Data Verification and Validation*;
PA-5102	PRS-ENM-5102	*Radiochemical Data Verification and Validation*;
PA-5105	PRS-ENM-5105	*Volatile and Semivolatile Data Verification and Validation*; and
ES-B-0811	PRS-ENM-0811	*Pesticide and PCB Data Verification and Validation.*
GEO-TEC-007	PRS-ENM-5007	*Data Management Coordination*
GEO-TEC-015	PRS-ENM-2300	*Subsurface Soil Sampling*
GEO-TEC-020	PRS-ENM-2300	*Surface Soil Sampling*
BJC-OS-1001	PRS-DOC-1009	*Records Management, Including Document Control*
PA-5003	PRS-ENM-5003	*Quality Assured Data – Paducah*
PA-5004	PRS-ENM-5004	*Sample Tracking, Laboratory Coordination and Sample Handling Guidance*
TR-9503	PRS-WSD-9503	*Shipping Samples from a Bechtel Jacobs Company Site*

Manufacturer's Instructions and Manuals		
		*Canberra Operating Manual for In Situ Gamma Spectroscopy**
		Millipore Envirogard© PCB Test Kit Standard Operating Procedure.
AD-MCS-7.10		*Calibration of GENIE-2000 Based HPGE GAMMA Spectroscopy System*
AD-MCS-7.12		*Operation of GENIE-2000 Based HPGE GAMMA Spectroscopy System*
Method 10050		***Immunoassay Method***
EPA 6200		***Field Portable X-Ray Fluorescence Spectrometry For The Determination Of Elemental Concentrations In Soil And Sediment***

* Hard copy available upon request.

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Environmental Monitoring	**PRS-ENM-0026**	**REV. NO. 0**
SUBJECT MATTER AREA: Data Verification and Validation	**PREPARER: Erica Hays**	**Page 1 of 38**
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REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of Paducah Remediation Services documentation. This document is a new procedure.	All

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1.0 PURPOSE

This procedure defines the minimum requirements, responsibilities, and methodology for the wet chemistry and miscellaneous analyses verification and validation processes for evaluating analytical data generated using industry standard measurement techniques.

This procedure is applicable to routine analyses for common wet chemistry parameters primarily in aqueous and soil/sediment matrices. Examples would include traditional water quality parameters (Sulfate, Chloride, Nitrate, etc.).

This method also applies to the verification and validation of data obtained for nonroutine miscellaneous analyses. Examples of these include determination of waste characteristics and other nonroutine analyses (e.g. flashpoint, coliform, etc.)

Specifications in this procedure should be incorporated into project documentation such as the Quality Assurance Project Plan, into contractual SOWs between the project and the analytical laboratories, and into contractual validation SOWs between the project and the organization chosen to validate the data. If validation is performed by individuals within the project, the SOW is not required, but a mechanism to specify validation requirements is recommended. This procedure shall be used as a baseline to create project-specific reports needed to perform wet chemistry and miscellaneous data verification and validation.

2.0 SCOPE

This procedure applies to wet chemistry and miscellaneous data verification and validation activities performed by PRS or its subcontractors.

3.0 OTHER DOCUMENTS NEEDED

- Analytical Method
- Laboratory SOW
- Data Validation Scope of Work

4.0 PRE-PERFORMANCE ACTIVITIES

Project Manager shall ensure that individuals who perform wet chemistry data verification and validation are knowledgeable of the latest version of this procedure before beginning any activities.

5.0 GENERAL INFORMATION

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by PRS.

5.1 Data Verification Requirements

5.1.1 Data verification is defined as a systematic process, performed either by the data generator (on-site or fixed-base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

5.1.2 If verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

5.1.3 Data verification, at the project level, may be conducted either by the data verifier or by the data validator, whichever expedites the review process. If verification is conducted independently of the validator, it included two activities. The first activity entails inventory of the data package to ensure compliance with the contract and SOW, in terms of the required deliverables. The second activity entails various checks of the data, the purpose of which is to alert the validator that a noncorrectable problem has occurred and that data may need qualification. If verification is conducted by the validator, the first activity is conducted similarly, but the second activity may result in prompt qualification of data.

5.1.4 Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an after-the-fact identification of noncorrectable deficiencies.

5.1.5 A verification report is written by the data verifier and takes, as input, the steps in this procedure that are listed as “Verification.” The data verifier does not qualify data if verification criteria are not met. Instead, they will document circumstances surrounding correctable and noncorrectable problems in the data package in the verification report. Following completion, the verification report will be transferred to the validator for possible qualification. The verification report must be in a standard format and must remain associated with the analytical data package. An example verification report, including all verification checks performed, is provided in Attachment B.

5.2 Analytical Data Validation Requirements

5.2.1 Analytical data validation, including laboratory data review, is defined as a systematic process, performed externally from the data generator, which applies a defined set of performance-based criteria to a body of data to determine the quality of reported results. <u>Data validation is not performed by the analytical laboratory</u>. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent and, if present, the level of uncertainty associated with the measurement. Analytical data validation for wet chemistry methods includes a technical review of the laboratory data package specified in the laboratory SOW. Validation incorporates an evaluation of sample custody,

sample handling and preparation, holding times, instrument calibration, instrument performance, batch quality control samples (e.g., Laboratory Control Sample), the identification and quantitation of target analytes, performance standards (e.g., surrogates, internal standards) and the effect QC performance and/or deficiencies have on the quality of analytical sample data.

5.2.2 A validation report that includes the results of validation activities must be completed by the validator and takes, as input, the verification report and the steps in this procedure that are listed as "Validation." An example validation report that includes all validation checks in this procedure is provided in Attachment C.

5.2.3 Data validation requires that personnel performing it have the appropriate level of training and experience to ensure data review and qualification is completed in a reasonable manner and in accordance with industry practices. Professional judgment may be required when performing data validation. Where professional judgment is used, resulting in either qualification of data or data left unqualified, the rationale for the selection of this path will be fully documented in the validation report. Documentation will include the following: citations from this procedure, other industry standards, and/or the literature demonstrating the reasonableness of the evaluation.

5.2.4 The actions described in this document must serve as the baseline for incorporation into project verification/validation activities. Project-specific procedures applying to analytical methods not covered in this document must be reviewed and approved prior to use.

5.2.5 Implementation of this procedure is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory SOW. Deliverable requirements specified in the analytical SOW must be consistent with the requirements of this procedure and with the Analytical Master Specifications of the PRS Terms and Conditions.

5.2.6 The validation SOW must be written consistent with the requirements and specifications of this procedure. The validation SOW is prepared by the Data Validation Lead and communicated from the project to the validation organization.

5.2.7 When qualification conditions are met, a qualifier shall be physically placed on the laboratory report form to ensure that data users, irrespective of time or organization, are aware of data limitations. Placement of the qualifier may be assigned by hand writing on the laboratory report form or electronically on laboratory deliverables. If data are not qualified during data validation, an equals sign ("=") shall be entered on the sample result.

5.2.8 A verification report (Attachment B or equivalent) and validation report (Attachment C or equivalent) must be completed for every

sample delivery group that undergoes data validation.

6.0 PROCEDURE

NOTE 1: Refer to Attachment D for qualifier descriptions. Refer to Attachment E for qualification guidance due to multiple quality deficiencies. Refer to Attachment F for a listing of relevant equations to use with this procedure.

NOTE 2: The Data Verifier performs all steps designated as "Data Verification" and the Data Validator performs all steps designated as "Data Validation."

6.1 Overall Implementation of Validation

The following is a step-by-step approach to implement analytical data verification and validation activities.

6.1.1 Validation Strategy and SOW Development

6.1.1.1 QA Specialist/Project Team shall develop validation strategy based on inputs identified through the DQO process. The project-specific sampling and analysis plan will define the DQOs and the framework for performing data validation.

6.1.1.2 QA Specialist/Project Team shall prepare a validation SOW to communicate verification and validation requirements to the organization performing the work.

6.1.2 Implementing Verification and Validation

Verifier and Validator shall implement verification and validation by completing the forms provided in Attachments B and C (or equivalent), respectively.

6.1.3 Custody of Samples and Sample Documentation

The COC form provides the basis for the traceability of project samples, by documenting the sample to its origin, as well as each step of the sampling, sample handling, and analysis process. By documenting personal accountability for samples, the COC is used to ensure that proper custody has been maintained from the time a sample is generated through its final disposition (cradle to grave). Any break in custody, as demonstrated by the series of signatures denoting sample holders, could jeopardize the defensibility of associated sample data.

While verification/validation cannot replicate the custody history of a sample (i.e., fully assure the sample truly has been in custody from the field to the final result), an evaluation of field notes, laboratory records, and the COCs provide the best available indicator of sample traceability. A sample is defined as being under proper

custody if any of the following conditions are met:

- The sample is within the possession of an authorized person (e.g. field personnel, laboratory personnel, etc.);
- The sample is within view of an authorized person;
- The sample was in an authorized person's possession and then was secured to prevent tampering; or
- The sample is placed in a designated secure area.

NOTE: Verification of sample documentation includes result report header checks for accuracy from the COC. If sample identity is in question, every attempt should be made to verify the true identity of each sample. When custody problems cannot be resolved, they will affect the defensibility of the sample.

6.1.3.1 Data Verification

The Data Verifier shall trace custody of all samples in the reporting batch from field sampling through receipt at the laboratory by reviewing the COCs. If the information is missing, the Data Verifier will seek to obtain field documentation from the sampler or contract laboratory to determine if the omission affects sample integrity. If there is a break in the signature chain on the COC, or other omissions in the custody record (e.g., date of sample collection, date of transfer to the laboratory, etc.), indicate the problem in the verification report.

6.1.3.2 Data Validation

Sample data that are not traceable through signature records on COCs or other sample record information demonstrating custody (e.g., laboratory logbooks) such that a complete custody history cannot be established, the Data Validator shall qualify associated results rejected "R."

6.2 Holding Time, Temperature, and Sample Preservation

Holding times have been established by the U.S. EPA to define the maximum period of time during which a sample remains representative of its sampling location. Holding times begin when a sample is collected in the field and are measured by determining the elapsed time from collection, through extraction (when applicable) and/or analysis. If the reported data is the result of a dilution, reinjection, or re-extraction and analysis, the result must have been generated within the prescribed holding time in order for the result to be considered definitive.

6.2.1 Deliverables

- Field Sampling Notes.
- Field COCs.
- Laboratory COCs.

- Laboratory Reports and/or raw data containing these: dates of collection; preparation; and analysis for all samples, dilutions, and re-extractions.

6.2.2 Criteria

Table 1.1 provides industry-accepted standards for sample containers, sample preservation, and holding times for routine analyses generally determined by wet chemical methods.

6.2.3 Data Verification

The Data Verifier shall verify the presence of the pertinent COC forms in laboratory deliverable. If information is missing, the Data Verifier will seek to obtain field documentation from the sampler and/or the contract laboratory, to determine if the omission affects sample integrity. Upon receipt, this information will be placed in the data package for delivery to the data validator

If missing information cannot be obtained or reconstructed from field notes, COCs, etc., the Data Verifier will note omitted information in verification report as noncorrectable.

Table 1.1 Wet Chemistry Parameters Holding Times, Containers, and Preservatives

Parameters	Containers	Preservatives	Holding Times
Specific Conductance	P, G	4 ± 2°C	28 days
Hardness	P, G	HNO_3 to pH<2	6 months
Residue			
Filterable (Total Suspended Solids)	P, G	4 ± 2°C	7 days
Nonfilterable (Total Dissolved Solids)	P, G	4 ± 2°C	7 days
Total	P, G	4 ± 2°C	7 days
Volatile	P, G	4 ± 2°C	7 days
Settleable Matter	P, G	4 ± 2°C	48 hours
Turbidity	P, G	4 ± 2°C	48 hours
Hexavalent chromium	P, G	4 ± 2°C	24 hours
Acidity and/or Alkalinity	P, G	4 ± 2°C	14 days
Bromide	P, G	4 ± 2°C	28 days
Chloride	P, G	4 ± 2°C	28 days
Chlorine	P, G	None required	Analyze immediately
Cyanides	P, G	4 ± 2°C NaOH to pH>12 0.6 g ascorbic acid*	14 days
Fluoride	P	4 ± 2°C	28 days
Iodide	P, G	4 ± 2°C	24 hours
Nitrogen			
Ammonia	P, G	4 ± 2°C, H_2SO_4 to pH<2	28 days
Kjeldahl, total	P, G	4 ± 2°C, H_2SO_4 to pH<2	28 days
Nitrate plus Nitrite	P, G	4 ± 2°C, H_2SO_4 to pH<2	28 days
Nitrate	P, G	4 ± 2°C	48 hours
Nitrite	P, G	4 ± 2°C	48 hours
Phosphorus			
Orthophosphate, dissolved	P, G	Filter on site; 4 ± 2°C	48 hours
Hydolyzable	P, G	4 ± 2°C, H_2SO_4 to pH<2	28 days
Total	P, G	4 ± 2°C, H_2SO_4 to pH<2	28 days
Total, dissolved	P, G	4 ± 2°C, H_2SO_4 to pH<2, Filter on site	24 hours
Silica	P, G	4 ± 2°C	28 days
Sulfate	P, G	4 ± 2°C	28 days
Sulfide	P, G	4 ± 2°C, add 2 mL zinc acetate plus NaOH to pH>9	7 days
Sulfite	P, G	None required	Analyze immediately
Total Organic Halides	P, G	Cool to 4° C, H_2SO_4 to pH<2	28 days
Uranium by TIMS	P, G	HCL or HNO_3 to pH<2	6 months
Biochemical Oxygen Demand	P, G	4 ± 2°C	48 hours
Chemical Oxygen Demand	P, G	4 ± 2°C, H_2SO_4 to pH<2	28 days
Oil & Grease	G only	4 ± 2°C, H_2SO_4 to pH<2	28 days
Total Organic Carbon	P, G	4 ± 2°C, H_2SO_4 or HCl to pH<2	28 days
Phenolics	G only	4 ± 2°C, H_2SO_4 to pH<2	28 days

* Only used in the presence of residual chlorine.
NOTE: The preservative for soils is cool to 4°C only.
P = Plastic G = Glass

6.2.4 Data Validation – Water / Liquid Holding Times

Review field and/or laboratory COC forms, field notes, laboratory report forms, and laboratory raw data, as necessary, to determine the elapsed time from sample collection to sample analysis.

If the elapsed time falls within the prescribed holding time, no actions will be taken and no qualification assigned. Place an "=" in the data qualification summary in the data validation report.

If holding times are exceeded by less than a factor of two (2), the Data Validator shall qualify detected compounds "J" and compounds < RDL "UJ."

If the holding time is exceeded by a factor of two (2) or more, qualify affected sample results > MDL with a "J" qualifier as estimated quantities and nondetect results "R" rejected due to gross holding time exceedance.

6.2.5 Data Validation – Soil Holding Times

Holding time criteria have not been definitively established for solid matrices. Established holding times for water generally are applied to soils to provide a measure of elapsed time from sample collection to analysis. If these criteria are applied to the validation of solid matrices, the validator must note the use of water holding times for the evaluation and provide justification for any qualifications assigned using water holding times in the validation report.

6.2.6 Data Validation - Temperature

If samples have exceeded temperature requirements, the validator must evaluate the effect on reported results. Depending on the magnitude of the temperature increase, results may or may not be adversely impacted. If prescribed sample receipt temperatures are exceeded (Table 1), qualify detected analytes "J" and nondetects "UJ."

If the temperature of samples grossly exceeds the limits prescribed in Table 1, the validator must evaluate the integrity of the reported concentrations. Consultation with the analytical method, other industry standard technical resources, and/or subject matter experts is advised if the temperature requirements are grossly exceeded to determine if sample / analyte degradation has occurred.

Data may be qualified "R" if the reviewer determines the effect of temperature exceedance has had a significant effect on the accuracy of reported sample results. Justification for "R" qualifiers must be provided in the validation report.

6.2.7 Data Validation – Sample Preservation

If samples have not been preserved properly in the field or have been stored improperly, qualify those sample results > RDL "J" as estimated quantities and results < RDL "R" as rejected values.

If improper storage also results in a loss of proper sample custody or increased sample temperatures, additional qualification may be required. Validators will evaluate such instances on a case-by-case basis. If a loss of custody or elevated sample temperature(s) coincides with improper storage, qualify affected sample results in accordance with sections 6.1.3.2 and/or 6.2.7 of this procedure.

6.3 Calibration

Instrument calibration or standardization is performed to ensure the instrument/analytical method is capable of producing quantitative data. Initial calibration/reagent standardization demonstrates that the instrument/ method is capable of acceptable performance at the beginning of the analytical run and of producing a linear calibration curve (if applicable for the method used). Initial and continuing calibration verifications demonstrate the method / instrument remains in control throughout sample analysis.

6.3.1 Deliverables

Specific deliverables will depend on the method and instrumentation used for analysis. Deliverables listed below will not be applicable to all wet chemical/miscellaneous methods (e.g., TSS, TDS). For methods where instrument calibration or reagent standardization is not employed to verify method accuracy, independent standards will be employed to provide a measure of method performance (e.g., laboratory control sample and/or matrix spike).

- Calibration Curve (where applicable)
- Reagent Standardization (where applicable)
- Initial Calibration Verification
- Continuing Calibration Verification
- Analysis Results
- Standard Preparation Log
- Analytical Run Log
- Raw data (required for confirmation).

6.3.2 Frequency

- Initial calibration or reagent standardization must be performed and shown to have acceptable linearity before any samples are analyzed.
- Initial calibration verification will be analyzed immediately following each successful instrument calibration / standardization.
- Continuing calibration verification will be analyzed prior to and following each group of 10 samples or following the last sample in a group – whichever is less.

6.3.3 Criteria

6.3.3.1 Initial Calibration

- At a minimum, initial calibration for instrumental methods will consist of a blank and three to five calibration standards bracketing the expected sample concentration(s).
- Initial Calibrations shall be generated daily for instrumental and most meter-type methods (e.g., spectrophotometric, colorimetric, turbidimetric, and ion chromatography) or each time the instrument is set-up for analysis – whichever is more frequent.
- The correlation coefficient for the calibration curve must be ≥ 0.995.
- For gravimetric methods (e.g., TSS, TDS, etc.), evidence of balance calibration must be provided with the laboratory deliverable to demonstrate proper calibration prior to measurement of sample weights.
- For titrimetric methods (e.g., alkalinity), documentation of titrant standardization must be provided as part of the laboratory deliverable to demonstrate the integrity of the titrant prior to analysis.
- If a pH meter is employed, the meter shall be calibrated with the two pH buffer solutions that bracket sample pH range.
- For nonroutine methods where calibration / standardization is not applicable, an independent check standard (initial calibration verification– below) will be employed prior to sample analysis to demonstrate the proper functioning of the method prior to sample analysis.

6.3.3.2 Initial Calibration Verification

For all routine wet chemistry and nonroutine methods, an ICV will be measured prior to the analysis of any project samples. The ICV will be prepared from a source other than that used to prepare calibration standards to demonstrate the method is acquiring accurate data.

The %R for the ICV will be within limits established by the laboratory or the analytical method as defined by the laboratory SOW. In the absence of

previously established limits, the %R for ICVs will be within 90-110%.

6.3.3.3 Continuing Calibration Verification

For all instrumental and most meter-type methods, CCVs will be analyzed prior to the analysis of any project samples and following each group of 10 samples or at the end of the sample group – whichever is more frequent. The CCV may be prepared from the same source as calibration standards. It will be prepared at the mid-point of the calibration curve to monitor instrument drift during analysis.

The %R for the CCV analysis will be within limits established by the laboratory or the analytical method as defined by the laboratory SOW. In the absence of previously established limits, 90-110% will be used to determine the accuracy of CCVs.

6.3.4 Data Verification

Data Verifier shall verify that appropriate documentation of the initial calibration and the initial and continuing calibration verifications have been provided in the data package. If any of the following occur, the Data Verifier shall contact the laboratory immediately to obtain the missing information:

- Evidence of initial calibration is not included in the laboratory deliverable;
- Frequency of calibration has not been satisfied; and/or
- Required numbers of calibration standards or required standard concentrations were not used.

Upon receipt, this information will be placed in the data package for delivery to the data validator

If these occurrences cannot be resolved with the analytical laboratory they are considered noncorrectable problems and shall be identified in this way in the data verification report. As they are contract compliance related, all such occurrences shall be communicated to Sample Manager and to the validator in the verification report.

6.3.5 Data Validation

If the instrument was not calibrated within the 24 hour period in which samples were analyzed, qualify sample results “R” rejected.

Verify that sample results were quantified within the linear range of the instrument and that the calibration standards bracket sample concentrations.

If the calibration curve does not bracket sample concentrations, determine if a linear range standard has been analyzed demonstrating that the upper linear concentration range of the instrument is capable of quantifying the analyte of interest. If no

such standard has been analyzed, the validator will review raw data and apply professional judgment to determine the effect on positive sample data. Nondetect results are unaffected by this condition. The following are provided as general guidance.

- If instrument responses exceed the calibration range, but show well resolved peaks (absent chart re-scaling), qualify results over the calibration range "J" estimated.

- If instrument responses grossly exceed the calibration range or saturate the instrument detector, qualify results "R" rejected.

If the correlation coefficient for the calibration curve is <0.995, qualify results >RDL "J," and results <RDL "UJ."

If the %R for the initial or continuing calibration verification is outside control limits, the following actions will be applied to qualify project data:

- If %R is 75-89% or 111-125%, qualify positive results as estimated, "J."
- If %R is within the range of 111-125%, nondetect results are acceptable.
- If %R is 75-89%, qualify nondetect results as estimated, "UJ."
- If %R is >125%, qualify positive results as unusable, "R;" nondetect results are acceptable.
- If %R is <75%, qualify all results (detects and nondetects) as unusable, "R."

6.4 Blanks

NOTE: Blank analysis may not be required for all wet chemical methods (i.e., titrimetric determinations). Refer to the specified analytical method in the laboratory SOW to determine if a method blank is required.

Blank analyses determine the existence and magnitude of contamination problems. A preparation or method blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation and analysis process. If required by the analytical method, instrument blanks are analyzed following the ICV and CCVs throughout the analytical run. If problems are observed with any blank, associated data must be evaluated carefully to determine whether contamination has occurred, if there is an inherent variability in method baseline, or if the problem is an isolated occurrence.

6.4.1 Deliverables

- Blank results.
- Sample preparation log and/or analytical run log.
- Raw data for each blank (required for confirmation).

6.4.2 Frequency

- Method blanks must be prepared and analyzed for each batch of 20 samples or less.
- The method blank must be of similar matrix in each SDG.
- If required by the analytical method, instrument blanks are analyzed following initial calibration and at a 10% frequency throughout the analytical run to follow CCVs.

6.4.3 Criteria

Target analytes should not be present in method or instrument blanks above the RDL.

6.4.4 Data Verification

Data Verifier shall verify the presence of the pertinent deliverables for blank analyses. If the required information is not present in the laboratory report, or if the frequency of analysis is not satisfied, the Data Verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If the information cannot be obtained, these occurrences are considered noncorrectable problems and will be identified as such in the data verification report. As this is a contract compliance issue, the occurrence should be communicated to the Sample Manager and the validator in the verification report.

6.4.5 Data Validation

Verify that results for the method blank and instrument blanks (if required) are reported accurately on the laboratory summary form from the raw data (level IV only). Data Validator shall qualify only if the deviation indicates an adverse effect on data quality.

All blanks associated with the case must be evaluated against the sample results in the case. However, qualification should be applied only to those samples directly related to the affected blank (if more than one method blank is used per case).

Any analyte that is reported in both blank and sample must be evaluated; however, if the same analyte is reported in the sample(s) and more than one blank, the sample(s) should be evaluated against the blank with the highest concentration of the analyte.

NOTE: Sample results must not be modified by subtracting blank values from sample concentrations.

- If the blank result has an absolute value less than or equal to the 2x the RDL, no qualification of data is necessary.

- If the reported blank result is greater than 2x the RDL, qualify all sample results greater than the reporting limit but <5X the amount in any blank as nondetects, "U." Sample results >5X the blank concentration require no qualification.

- If the blank result is negative, and the absolute value is greater than 2x the RDL, all associated samples must be carefully examined. Sample results reported as positive, but <5X the absolute value of the blank shall be qualified "J," and sample results reported as nondetects, will be qualified "UJ."

- If gross contamination is present, the positive results must be evaluated carefully to determine if they are false positives. When gross contamination is observed in the method or instrument blank, qualify positive results < 10x the blank value "R." Results > 10x the blank value should be qualified "J." Nondetects are unaffected by this condition.

6.5 Laboratory Control Sample

The LCS serves to monitor the overall performance of all steps in the analysis, including sample preparation and instrumental analysis.

NOTE: In very limited cases, an LCS is not be required (e.g., coliform). However, the overwhelming majority of wet-chemical / nonroutine methods require an LCS. Refer to the specific analytical method for guidance.

6.5.1 Deliverables

- LCS results
- Sample preparation log
- Analytical run log
- Raw Data (required for confirmation)

6.5.2 Frequency

An LCS must be prepared and analyzed with each batch of samples numbering 20 or less requiring preparation (i.e., digestion, filtration, extraction) before analysis. For methods not requiring sample preparation, an independent standard such as an ICV may be substituted for the LCS to monitor instrument performance.

6.5.3 Criteria

LCS percent recoveries must be within control limits specified by the analytical method, the standard supplier, or the laboratory. This will be specified in the laboratory SOW.

If laboratory-generated control limits or vendor-supplied limits are not

provided, control limits for LCS percent recovery of 80-120% shall be used.

6.5.4 Data Verification

The data verifier shall verify the presence of LCS results. If results are omitted from the laboratory report, the Data Verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If LCS analysis was required, but not performed, this is considered a noncorrectable problem, and shall be indicated in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the Sample Manager and the validator in the data verification report.

6.5.5 Data Validation

- If LCS recovery falls within the range of 50-79% or >120%, qualify detected results as estimated, “J.”
- If LCS recovery is >120%, nondetect results are acceptable and no qualification is required.
- If LCS recovery falls within the range of 50-79%, qualify positive results “J” and nondetect results, “UJ.”
- If LCS recovery results are <50%, qualify positive results as estimated, “J,” and nondetect results as unusable, “R.”

6.6 Matrix Spike/Matrix Spike Duplicate or Matrix Spike/Duplicate

MS/MSD data are generated to determine the precision and accuracy of the analytical method in the specific sample matrices. They provide a sample/project-specific measure of the method’s ability to recover target analytes under real sample conditions.

NOTE: MS/MSD or MS/Duplicate analyses may not be required for all wet chemical / nonroutine methods (e.g., gravimetric, titrimetric, coliform, ignitability, and others). Refer to the specific analytical method to determine the appropriateness of a MS / MSD.

6.6.1 Deliverables

- MS/MSD (or MS/Duplicate) results
- Sample preparation log
- Analytical run log
- Raw Data (required for confirmation)

6.6.2 Frequency

MS/MSD or MS/Duplicate samples must be analyzed at a frequency of at least one pair per 20 field samples, and they must be prepared from the same or similar matrix for the samples they accompany. If multiple matrices are prepared together (e.g., water and soil) by the laboratory, individual MS/MSDs must be prepared to reflect each

matrix type.

6.6.3 Criteria

MS/MSD recoveries must be within control limits specified by the analytical method or those generated by the laboratory. This will be defined in the laboratory SOW. Absent predefined control limits 75-125% shall be used.

Precision for duplicates or MSDs is measured in terms of RPD. Duplicate or MSD precision must be within control limits specified by the analytical method or those generated by the laboratory. This will be defined in the laboratory SOW. In the absence of predefined control limits, +/- 20% RPD for aqueous samples, and ±35% RPD for soil / solid matrices shall be used.

6.6.4 Data Verification

Data verifier shall verify that MS/MSD or MS/Duplicate samples were analyzed at the required frequency and that results are provided for each sample. If results are omitted from the laboratory report, the Data Verifier will contact the laboratory to obtain the omitted information. Upon receipt, this information will be placed in the data package for delivery to the data validator.

If results cannot be obtained or the frequency of analysis is not satisfied, these occurrences are considered noncorrectable problems and will be identified as such in the data verification report. As this is a contract compliance issue, the occurrence will be communicated to the Sample Manager and validator in the data verification report.

The laboratory is required to use a natural sample when preparing and analyzing MS/MSDs. If a field blank has been used for the MS/MSD, the Data Verifier will consult with the Sample Manager to determine an appropriate course of action. Samples may require reanalysis to ensure project data is not affected by this action. If not, the issue will be identified as uncorrectable in the data verification report and feedback will be provided to project personnel and the laboratory to ensure this is not repeated.

6.6.5 Data Validation

If MS/MSD or MS/Duplicate analysis was required, but not performed, qualify only if the deviation indicates an adverse effect on data quality. Occasionally, limited sample volumes prevent the preparation and analysis of MS/MSDs. In these cases, it is common practice for the laboratory SOW to allow for the analysis of an LCS / LCS duplicate pair as a substitute to provide an evaluation of precision in the measurable range of the method.

In the absence of either the MS/MSD or LCS/LCSD, it is unlikely that

a complete evaluation of method precision and accuracy can be completed. In this case, at a minimum, sample results should be considered estimated quantities due to the inability of data users to fully determine the quality of the reported results. Affected positive results shall be qualified "J" and nondetects "UJ" unless other quality deficiencies are observed.

Recalculate one MS recovery from raw data. Equation F1 in Attachment F is used to calculate MS percent recovery (level IV only). If the MS/MSD or LCS/LCSD have been provided and recovery difficulties have been experienced, the following guidance shall be used for evaluating accuracy:

- If poor spike recovery occurs in a sample whose concentration is > 4x the spiked amount, no qualification is warranted.
- If MS recoveries are > 125%, qualify detected analytes as estimated, "J." Nondetects do not require qualification.
- If MS recoveries are > 30% and < 75%, qualify detected analytes as estimated, "J," and nondetects as estimated, "UJ."
- If MS recoveries are < 30%, qualify detections "J" estimated and nondetects "R" rejected.

If poor duplicate or MS/MSD precision is observed, the following guidance shall be used:

- If the RPD for water / liquid duplicate or MS/MSD is > 20% and < 35%, qualify associated detections "J" and nondetects "UJ."
- If the RPD for water / liquid duplicate or MS/MSD is > 35% qualify all associated sample results "R."
- If the RPD for soil / solid matrices > 35% and < 50%, qualify associated detections "J" and nondetects "UJ."
- If the RPD for soil / solid matrices > 50%, qualify all associated results "R."

6.7 Sample Result Verification

If the laboratory has a high rate of manual transcription in generation of sample results, the project team may choose to recalculate manually sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contact the laboratory to resolve the problem. If calculations have been determined to be performed incorrectly, such that reported results are incorrect, the Data Validator will make corrections manually during validation. If consultation with the laboratory cannot resolve calculation anomalies, the data validator will use professional judgment to determine the effect on the data set. Data may be qualified "J" estimated or "R" rejected depending on the severity of the issue and the extent to which it impacts the data.

If results are to be manually recalculated from raw data, the following strategy is recommended:

- Examine raw data for anomalies.

- Verify from raw data, two detected and nondetected results for each analyte and/or method of analysis.
- Confirm the initial and final sample volumes/weights used for sample analysis from sample preparation logs.
- All analyses must fall within the instrument calibration range. If outside, confirm that the sample has been diluted and reanalyzed and that results are corrected for dilution factor(s).

7.0 RECORDS

Generate and maintain all records in accordance with BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

- The data verification report
- The data validation report
- Copies of qualified or unqualified results reports

8.0 SOURCE DOCUMENTS

- EPA QA/G-4, September 2004. *Guidance for the Data Quality Objectives Process*.
- EPA QA/R-5, March 2001, *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations*
- EPA-600/4-79-020, March 1983. *Methods for Chemical Analysis of Water and Wastes.*
- EPA-540/R-04/004. October 2004. *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review.*
- SW-846 third edition, November 1986. *USEPA Test Methods for Evaluating Solid Waste.* Revisions through Update III.

Attachments A
DEFINITIONS/ACRONYMS
Page 1 of 3

DEFINITIONS

NOTE 1: Qualifier definitions are listed in Attachment D.

NOTE 2: In this procedure, the words “shall” and “must” are used to denote a requirement; the word “should” is used to denote a recommendation; and the word “may” is used to denote permission (neither a requirement nor a recommendation). In conformance to this procedure, all steps shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

AFFECTED SAMPLE RESULT – A sample result is considered to be affected when it is significantly influenced by a quality deficiency and is qualified accordingly through analytical data validation.

ANALYTICAL DATA VALIDATION – Analytical data validation is a systematic process, performed independently from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

ANALYTICAL DATA VERIFICATION – Analytical data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract that is performed either by the data generator or by an entity independent to the data generator.

BATCH – A batch is a group of samples prepared at the same time in the same location using the same method, not to exceed 20 samples of similar matrix.

CASE – A finite, usually predetermined number of samples, that have been collected over a given time period from a particular site. A case consists of one or more sample delivery groups.

CHAIN OF CUSTODY – The history of the transfer of samples from the time of sample acquisition through archival and disposal of samples. Chain-of-Custody documentation is required as evidence of sample integrity.

CONTINUING CALIBRATION VERIFICATION – A standard solution analyzed at a specified frequency during an analytical run to assure continued validity of the calibration curve.

CORRECTABLE PROBLEM – Correctable problems are deficiencies within data packages that may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation. Correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems revealed during validation are those deficiencies with analyses that can be solved either by a second preparation and/or by analysis of a sample.

Attachments A
DEFINITIONS/ACRONYMS
Page 2 of 3

DATA QUALITY OBJECTIVES – DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO Process that specify the study objectives, domain, limitations, the most appropriate type of data to collect, and specify the levels of decision error that will be acceptable for the decision.

DATA QUALITY OBJECTIVES PROCESS – The DQO Process is a quality management tool based on the scientific method and developed by EPA to facilitate the planning of environmental data collection activities. The DQO Process enables planners to focus their planning efforts by specifying the use of the data (the decision), the decision criteria (action level), and the decision maker's acceptable decision error rates.

HOLDING TIME – Holding time, as described in this procedure, is defined as the period of time between sample collection and sample activity determination.

LABORATORY CONTROL SAMPLE – The LCS is a control sample of known composition. Aqueous and solid LCS are analyzed using the same preparation, reagents, and method employed for field samples.

LABORATORY DUPLICATE – The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

MATRIX SPIKE – The matrix spike is a split of a field-originating analytical sample in which one half of the split is spiked with a known amount of radionuclide of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation

NONCORRECTABLE PROBLEM – Noncorrectable problems are deficiencies within data package that preclude the evaluation of data quality by predefined criteria. Noncorrectable problems may be revealed during both data verification and data validation.

PREPARATION BATCH – A preparation batch is a group of sample aliquots prepared together at the same time using the same method and related to the same quality control samples.

QUALITY-INDICATOR SAMPLE – Quality-indicator samples are those samples made ready in the laboratory that provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

REQUIRED DETECTION LIMIT – The RDL is a contractually specified detection limit that, under typical analytical circumstances, should be achievable.

SAMPLE DELIVERY GROUP – An SDG is defined by one of the following, whichever occurs first: 1) Case of field samples; 2) Each 20 field samples within a case; 3) Each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the SDG.

Attachment A
DEFINITIONS/ACRONYMS
Page 3 of 3

SAMPLE RESULT – A sample result, as described in this procedure, is a numeric denotation of the concentration, amount, or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

STATEMENT OF WORK – The validation SOW is a document prepared to function as the mechanism by which validation requirements are communicated from the project to the validation organization.

TURN-AROUND TIME – Turn-around time is contractually specified as the amount of time that elapses between laboratory receipt of the raw samples and subsequent data receipt by the client.

VALIDATION QUALIFIER – A qualifier is an alphabetic character physically or electronically associated with a discrete sample result during validation due to a data quality deficiency, which provides guidance in data usability.

VALIDATION STATEMENT OF WORK – The validation SOW is a document prepared to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

ACRONYMS

%R – percent recovery
CCV – Continuing Calibration Verification
COC – Chain of Custody
DQO – Data Quality Objective
EPA – Environmental Protection Agency
ICV – initial calibration verification
LCS – Laboratory Control Sample
MS/MSD – Matrix Spike/Matrix Spike Duplicate
PRS – Paducah Remediation Services, LLC
QA – Quality Assurance
QC – Quality Control
RDL – Required Detection Limit
RPD – Relative Percent Difference
SDG – Sample Delivery Group
SOW – Statement of Work

Attachment B
WET CHEMISTRY DATA VERIFICATION REPORT
Page 1 of 3

(Pages 2 through 3 contain a full size copy of the report and may be used directly. The pages have no headers.)

Wet Chemistry Data Verification Report

Project Name: _____

Sample Delivery Group #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW#: _____

This checklist, or equivalent, is to be used for verification of Wet Chemistry data packages, consistent with procedure PRS-ENM-0026.

Verified by: _____ Date: _____

Wet Chemistry Data Verification Report

	YES	NO	NA
Custody of Samples			
1. Are samples traceable through inspection of signature records on field and laboratory COCs?	_____	_____	_____
Action: For any samples not traceable upon inspection of COCs, indicate the identities below and where the break in traceability occurred.			
Calibration			
2. Has initial calibration been performed prior to analysis of analytical samples?	_____	_____	_____
3. Are initial and continuing calibration results included in the data package?	_____	_____	_____
4. Have frequency requirements for calibration been satisfied?	_____	_____	_____
Blanks			
5. Are results for method and/or instrument blanks included in the data package?	_____	_____	_____
6. Have frequency requirements for the method blank been satisfied?	_____	_____	_____
Matrix Spike/Matrix Spike Duplicates or MS/Duplicate			
7. Are MS/MSD or MS/Duplicate results included in the data package?	_____	_____	_____
8. Have frequency requirements for the MS/MSD been satisfied?	_____	_____	_____
9. Have field blanks been used for MS/MSD or MS/Duplicate analysis?	_____	_____	_____
10. Are RPD results reported for MS/MSD or MS/Duplicate analysis?	_____	_____	_____
Laboratory Control Samples			
11. Are LCS results included in the data package?	_____	_____	_____
12. Have frequency requirements for the LCS been satisfied?	_____	_____	_____

Comments: _____

_____

_____

Attachment C
WET CHEMISTRY DATA VALIDATION REPORT
Page 1 of 9

(Pages 2 through 9 contain a full size copy of the report and may be used directly. The pages have no headers.)

Wet Chemistry Data Validation Report

Project Name: _____

Sample Delivery Group #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW#: _____

This checklist, or equivalent, is to be used for validation of Wet Chemistry data packages, consistent with procedure PRS-ENM-0026. This checklist should be used only if the Wet Chemistry Data Verification Report has been completed.

Instructions: If criteria are not met in this checklist, follow qualification provided in the "Q" column. If necessary, additional codes may be placed. Sections titled "Raw Data Confirmation" need only be performed if necessary, as determined through the DQO process.

Validated by: _____ Date: _____

Custody of Samples

Validation Step
1. Are samples traceable? Yes ☐ No ☐

Action: For any samples not traceable upon inspection of verification report and chains of custody, indicate the identities below and where the break in traceability occurred. Qualify samples not traceable "R." Indicate which samples were affected below.

Qualified Sample ID:

Wet Chemistry Data Validation Report

Holding Times and Sample Preservation

Validation Step		Qualification Guidance	
		Detects	Nondetects
1. Were all samples analyzed within the appropriate holding time?	Yes ☐ No ☐ NA ☐	J	UJ/R
2. Were all samples preserved properly?	Yes ☐ No ☐ NA ☐	J	UJ

Action: Indicate affected samples below.

Sample ID:	Holding Time Exceedance (Days):	Improper Preservation:	Qualification

Comments:

Calibration

Validation Step		Qualification Guidance	
		Detects	Nondetects
1. Was the instrument calibrated at the appropriate frequency?	Yes ☐ No ☐ NA ☐	*	*
2. Was the minimum number of standards used in calibrating the instrument?	Yes ☐ No ☐ NA ☐	*	*
3. Was the regression ≥ 0.995?	Yes ☐ No ☐ NA ☐	J	UJ
4. Were continuing calibration recoveries within control limits? **	Yes ☐ No ☐ NA ☐	J/R	UJ/R
• Is %R > upper control limit?		J	NA
• Is %R < lower control limit?		J	UJ

* Qualify only if the deviation indicates an adverse effect on data quality.
** See procedure for further guidance if no laboratory criteria are provided.

Action: Indicate affected samples below.

Sample ID: Reason for Qualification:

Comments:

Method Blanks

Validation Step		Qualification Guidance	
		Detects	Nondetects
1. Were method blanks and instrument blanks (if required) prepared and/or analyzed at the appropriate frequency?	Yes ☐ No ☐ NA ☐	*	*
2. Were sample results verified as uncorrected for blank concentrations?	Yes ☐ No ☐ NA ☐	J	N/A
3. Are all sample results >CRQL and >5** times the blank result?	Yes ☐ No ☐ NA ☐		
• Sample result is >CRQL and <5X the blank result		U	NA
• Sample result is <CRQL and <5X the blank result		U	NA
• Gross contamination		R	NA
4. Were negative concentrations in blanks evaluated?	Yes ☐ No ☐ NA ☐	N/A	U
5. Was the presence of blank contaminants confirmed from raw data? (Applies to Level D data only.)	Yes ☐ No ☐ NA ☐	**	**

* Qualify only if the deviation indicates an adverse effect on data quality.
** Use professional judgment in qualifying data.

Action: Indicate affected samples below.

Sample ID:	Reason for Qualification:

Comments:

Laboratory Control Samples

Validation Step		Qualification Guidance	
		Detects	Nondetects
1. Have LCS results been included in the data package?	Yes ☐ No ☐ NA ☐	*	*
2. Was the LCS prepared and analyzed at the appropriate frequency?	Yes ☐ No ☐ NA ☐	*	*
3. Were percent recoveries within acceptance limits? **	Yes ☐ No ☐ NA ☐	J/R	UJ/R
• Is %R > upper control limit?		J	NA
• Is %R < lower control limit?		J	UJ/R

* Qualify only if the deviation indicates an adverse effect on data quality.
** See procedure for guidance if no laboratory criteria are provided.

Action: Indicate affected samples below.

Sample ID:	Reason for Qualification:

Comments:

Wet Chemistry Data Validation Report

Matrix Spike/Matrix Spike Duplicate (MS/MSD) or MS/Duplicate

Validation Step		Qualification Guidance	
		Detects	Nondetects
1. Have MS/MSD or MS/Duplicate results been included in the data package?	Yes ☐ No ☐ NA ☐	---	---
2. Were the MS/MSD or MS/Duplicate analyzed at the appropriate frequency?*	Yes ☐ No ☐ NA ☐	---	---
3. Are MS and MSD percent recoveries within acceptance criteria? **	Yes ☐ No ☐ NA ☐	J/R	UJ/R
• Is %R > upper control limit?		J	NA
• Is %R < lower control limit?		J	UJ/R
4. Are all MS/MSD (or sample and duplicate) RPDs within control criteria? **	Yes ☐ No ☐ NA ☐	J	UJ

* Qualify only if the deviation indicates an adverse effect on data quality.

** Qualify only if other QC data in the SDG is outside established criteria.

Action: Indicate affected samples below.

Sample ID:	Reason for Qualification:

Comments:

Sample Result Verification

	Validation Step
	For the following evaluation, some qualification of sample data may be possible. For contractual noncompliance, a validation code is placed if the occurrence is noncorrectable.
1.	For each SDG, recalculate 2 detected and 2 nondetected results for each wet chemistry method from the raw data. (Applies to Level D data only.)
2.	Did recalculation confirm reported results? Yes ☐ No ☐ If not, increase the frequency of recalculations until adequate confidence is gained in the reported results. (Applies to Level D data only)
3.	Were reported results within the calibration range of the instrument? Yes ☐ No ☐
4.	Were results from diluted samples corrected for the dilution factor? Yes ☐ No ☐

Action: Indicate instances of manual calculations not confirming reported results.

Where samples have been reanalyzed and both analyses are included in the data package, indicate on the laboratory reporting forms which results are the most reliable.

Sample ID:	Reason for Qualification:

Comments:

Attachment D
DATA VALIDATION QUALIFIERS, AND QUALIFICATION CODES

Wet Chemistry Data

U Analyte compound or nuclide considered not detected above the reported detection limit.

J Analyte compound or nuclide identified; the associated numerical value is approximated.

UJ Analyte, compound, or nuclide not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.

R Result is not usable for its intended purpose.

= “Equals” sign, indicates that no qualifier is necessary.

Attachment E
QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES
Page 1 of 1

Guidance for Data Qualification Due to Multiple Quality Deficiencies

This attachment provides guidance in the qualification of data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on potential effect on sample data. The effect of quality deficiencies may be applicable to only a single sample or to all samples within the reporting batch. A validation qualifier should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

The following is a listing of data quality indicators and the probable effects on sample data.

Data Quality Indicator	Effect on Sample Data
Standard curve correlation coefficient	Quantitative uncertainty
Continuing calibration verification	Positive or negative bias
Method blank	Positive bias
Laboratory control sample	Positive or negative bias and precision
Matrix spike/matrix spike duplicate	Positive or negative bias and precision

In the instance of multiple quality deficiencies the validation qualifier should be placed consistent with the acceptable level of uncertainty associated with the intended use of the data. The validation SOW should provide a summary of the intended use(s) of the data. (e.g., risk assessment, fate and transport modeling, waste management) to facilitate appropriate placement of validation qualifiers.

Attachment F
RULES, CALCULATIONS, AND EQUATIONS

Rounding Rules

1. In a series of calculations, carry the extra digits through to the final result, and then round off.

2. If the digit to be removed is less than 5, the preceding digit stays the same.

3. If the digit to be removed is equal to or greater than 5, the preceding digit is increased by 1.

Calculations/Equations

F.1 Matrix spike percent recovery

$$Conc_{MS} = \frac{SSR - SR}{SA} x100$$

where: SSR = Spiked Sample Recovery
SR = Sample Result
SA = Spike Added

F.2 Relative Percent Difference

$$RPD = \frac{|R_1 - R_2|}{\overline{X}_{R_1,R_2}} x100$$

where: R1 = first sample value (original)
R2 = second sample value (duplicate)

F.3 Laboratory Control Sample and Continuing Calibration Verification Percent Recovery

$$\%R = \frac{FOUND}{TRUE} x100$$

where: FOUND = Concentration (in ug/L for aqueous; mg/kg for solid) of each analyte measured in the analysis of LCS or CCV Solution.

TRUE = Concentration (in ug/L for aqueous; mg/kg for solid) of each analyte in the LCS or CCV source.

F.4 Sample Results - Refer to the specific analytical method for equations for calculation of sample results for soil and water.

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date April 24, 2006

PROCEDURE TO BE TRANSITIONED:

EH-4516

Functional	Radioactive Contamination Control and Monitoring
Procedure Type (Company, Functional, Project, Subcontractor)	(Procedure Number and Title)
August 31, 2004	6
(Procedure Date)	(Rev. Number)

Reason for Changes: Transition BJC document to PRS interim document

SECTION	DESCRIPTION OF CHANGE
Entire Document	Change "Bechtel Jacobs Company LLC" to "Paducah Remediation Services LLC"
Entire Document	Change "BJC" to "PRS", except in procedure numbers/titles
Scope	Change "East Tennessee Technology Park, Oak Ridge National Laboratory, the Y-12 Plantand any other sites and facilities" to "Paducah Gaseous Diffusion Plant"
Entire Document	Replace BJC positions/titles with PRS positions/titles as indicated on attached matrix/crosswalk

Initiator/Originator: William R Chase — 04/05/06
(Print Name / Sign Name) — Date

REVIEWED BY: Neil C. Kiely — 4-6-06
Discipline / Task Manager — Date

APPROVED BY: CARLESTON EDMUNDS — 4/08/06
Project Manager — Date

BJC to PRS Position/Title Matrix

BJC Positions	PRS Position
Alternate Source Custodian	Technical Program Specialist
BJC General Manager	PRS General Manager
BJC President	PRS President
Control Technicians	Radiation Control Technician or Dosimetry Technician
Deputy General Manager	Manager of Projects
Dosimetrist	Technical Program Specialist
Dosimetry Program Specialist	Technical Program Specialist
Dosimetry Specialist	Technical Program Specialist
ES&H Manager	ES&H Manager
ESH&Q Assessment Coordinator	ESH&Q Manager
Facility Manager	Facility Manager
Female General Employee	Female General Employee
Field Engineer	Field Engineer
Field Operations Manager	Field Operations Manager
Field Radiological Engineer	Radiological Engineer
First Line Manager	Supervisor
Functional Manager	Department Manager (Ex. RPPM for RADCON)
Health Physicist	Radiological Engineer
Instrumentation Supervisor	Technical Program Specialist for RADCON
Junior Radiological Control Technician	Junior Radiological Control Technician
Manager of Projects	Manager of Projects
On-scene Commander (Emergency Squad)	EOC Director
Operations Support Manager	Project Manager
Plant Shift Superintendent (USEC)	Plant Shift Superintendent (USEC)
Project Health Physicist	Radiation Protection Program Manager or designee
Project Line Management	Project Line Management
Project Manger	Project Manger
Project RADCON Manager	Radiation Protection Program Manager
Purchasing Agent	Subcontract Administrator
RADCON Internal Audit Coordinator (RAIC)	Technical Program Specialist
RADCON Manager	Radiation Protection Program Manager
RADCON Subcontractor	RADCON
RADCON Technician	Radiation Control Technician
Radiation Control Organization	RADCON
Radiation Control Technician	Radiation Control Technician
Radiation Control Technician Supervisor	Radiation Control Technician Supervisor
Radiation-generating Device Facility Manager	Facility Manager
Radiation-generating Device Operator	Radiation-generating Device Operator
Radiographers	Radiographers
Radiological Control Manager	Radiation Protection Program Manager
Radiological Control Officer	Radiation Protection Program Manager
Radiological Engineer	Radiological Engineer
Radiological Worker	Radiological Worker
Receiving Agent	Receiving Person
RWP User	RWP User
Safety Advocate	ESH&Q Safety Specialist
Senior Radiological Control Technician	Senior Radiological Control Technician
Site Dosimetry Coordinator	Technical Program Specialist
Site Dosimetry Manager	Technical Program Specialist
Source Custodian	Technical Program Specialist
Subcontract Coordinator	Subcontract Administrator
Subcontract Technical Representative	Project Manager
Subcontractor Assigned ES&H Representative	Assigned ES&H Representative
Supervisor	Supervisor
Supervisor of Declared Pregnant Worker	Supervisor of Declared Pregnant Worker
Technical Support Manager	Technical Program Specialist for RADCON
Training Manager	Training Manager
Work Group Supervisor	Supervisor

OWNER: Radiological Controls	EH-4516	REV. NO. 6
SUBJECT AREA: Monitoring; Survey	EFFECTIVE DATE: 8/31/04	Page 1 of 15
DOC TYPE: Procedure	PREPARER/POC: Michael W. Stafford	
TITLE: RADIOACTIVE CONTAMINATION CONTROL AND MONITORING	APPROVED BY/DATE: Mike Stafford 8/6/04 [Approval Signature On File]	

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Attachment B EXAMPLE CONTAMINATION SURVEY FORM 13

CAUTION

This procedure contains commitments specific to Title 10, Code of Federal Regulations, Part 835, *Occupational Radiation Protection,* and the Bechtel Jacobs Company LLC Issues and Corrective Action Tracking System (I/CATS). The source of the specific commitment follows the procedural step in brackets (e.g., [§835.1102(c)] or [I/CATS Action ID 8867]).

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Non-intent field change. Clarified how to document and review surveys. Replaces SH-B-4012, Rev. 3, *Radioactive Contamination Control and Monitoring*.	9 and 10
1	Intent Change. Shifts responsibilities from BJC to BJC RADCON subcontractor.	2, 5, 7, 8
2	Non-Intent Change. Deleted Section I, Contamination Survey Techniques" in response to I/CATS Action 4057. This section was determined to be unnecessary and subject to misinterpretation. Previous Section J is now Section I.	8, 9
3	Non-Intent Change. Added a note prior to Section H.1 which reads as follows: "It is not the intent of Survey and Release Plans to substitute for Sampling and Analysis Plans or to be used to support release/disposal decisions based on volumetric contamination. Survey and Release Plans are used when surface contamination values in Attachment A apply."	7
4	Non-Intent Change. Added references to Other Documents Needed and added Note to Section I.6. Removed Training Section per BJC-PQ-1107, Rev. 3.	2, 9
5	Intent Change. Revised to delete references to specific sites. Deleted requirement for annual review of monitoring frequencies. Changed personnel contamination control Note to a requirement. Added reference to Chapter 6 of EH-4500. Added requirements for handling and counting smears in the field. Added clarifying note concerning the 80% ACL. Included PHP as performer associated with custodial justifications. Provided exception for survey documentation time-frame if awaiting laboratory results. Addressed corrective actions in I/CATS Action IDs 8867 and 9067.	3 - 11
6	Intent Change. Clarified Scope. Changed performers from subcontractor to BJC. Deleted references to CCZs.	3, 4, 6, 8, 9, 14

PURPOSE

This procedure defines the contamination control and monitoring requirements for Bechtel Jacobs Company LLC (BJC). The requirements in this procedure are the minimum steps necessary for compliance with the applicable regulatory requirements for radioactive contamination control and monitoring and for the release of materials and equipment from radiological areas as specified in 10 CFR 835, and conformance with mandatory contractual and administrative requirements. This procedure also controls unrestricted release of materials and equipment from Controlled Areas.

SCOPE

This procedure applies to radioactive contamination control and monitoring conducted under the BJC Radiological Protection Program at the East Tennessee Technology Park, Oak Ridge National Laboratory, Y-12 National Security Complex and at any sites or facilities where BJC conducts radiological operations for the U.S. Department of Energy (DOE) Oak Ridge Operations Office.

OTHER DOCUMENTS NEEDED

- BJC-EH-4006, *Embryo/Fetus Protection*
- BJC-EH-4030, *Radiological Area Entry Control*
- BJC-EH-4035, *Radiation Safety Training*
- BJC-GM-520, *Documenting Radiation/Contamination Surveys*
- Directive 08, *P/QA Review of Unconditional Release Surveys*
- EH-4500, *Radiological Control Operations Guide for the Environmental Management and Enrichment Facilities at Oak Ridge, Tennessee, Portsmouth, Ohio, and Paducah, Kentucky*
- EH-4511, *External Dosimetry*
- EH-4512, *Internal Dosimetry*
- EH-4513, *Workplace Monitoring*
- EH-4517, *Posting and Labeling*
- EH-4518, *Radiation Protection Program Records*
- SH-B-GLOS, *Radiation Protection Glossary*

WHAT TO DO

A. Surface Contamination Values

Radiological Controls (RADCON) Technician (RCT)

1. Any surface on which contamination levels exceed the values specified in Attachment A, "Surface Contamination Values," is considered contaminated and shall be [§835.1102(b)]:

a. Posted and/or labeled in accordance with EH-4517.

b. Controlled in a manner commensurate with the physical and chemical characteristics of the contaminant, the radionuclides present, and the fixed and removable contamination levels.

B. Fixed Contamination Areas

NOTE: Entry controls as required in §835.501 and posting as required in §835.603 are not required for fixed contamination areas meeting the following requirements.

Project Health Physicist (PHP)

1. Control areas accessible to individuals where the measured total surface contamination levels exceed, but the removable surface contamination levels are less than the corresponding surface contamination values in Attachment A, as follows [§835.1102(c)]:

a. The area shall be routinely monitored at a determined frequency to ensure the removable surface level remains below the removable surface contamination values specified in Attachment A [§835.1102(c)(1)].

b. The area shall be conspicuously marked to warn individuals of the contaminated status [§835.1102(c)(2)].

C. Area Control

Project Health Physicist (PHP)

1. Maintain appropriate controls that prevent the inadvertent transfer of removable contamination to locations outside of radiological areas under normal operating conditions [§835.1102(a)]. Verify these controls by documented inspection.

2. Use the following measures, as applicable, to prevent the spread of contamination across the boundary of Contamination Areas (CA), High Contamination Areas (HCA), or Airborne Radioactivity Areas (ARA):

a. Use barriers to enclose the area.

b. Mark and secure items such as hoses and cords that cross the boundary.

c. Control and direct airflow from areas of lesser to areas of greater removable contamination, whenever practicable.

d. Use engineered controls and containment devices such as glove bags, glove boxes, and tents.

D. Exit Control

NOTE: Use of a step-off pad may present slipping or tripping hazards when used in wet areas or on uneven terrain. In such case the Site Manager or designee may waive the requirement for a step-off pad. Step-off pads should not be used outdoors.

RCT

1. Include the following at exit points from frequently accessed CAs and ARAs:

- A step-off pad placed immediately outside the exit point, contiguous with the area boundary, and maintained free of radioactive contamination;
- Containers near the step-off pad inside the area boundary for the collection of protective clothing, equipment, and radioactive waste; and
- Contamination monitoring equipment placed as close to the step-off pad as background radiation levels and environmental conditions permit.

2. Have either the same exit controls (as frequently accessed areas) for infrequently accessed CAs and ARAs or provide arrangements for frisking and waste disposal (no step-off pad required). Examples include abandoned, unoccupied and deactivated facilities.

3. Use multiple step-off pads, as appropriate, at the exits of HCAs to control and limit the spread of contamination. The following controls apply when multiple step-off pads are used:

- The inner step-off pad shall be placed immediately outside the HCA, but still within the CA;
- The worker shall remove the outer clothing before stepping on the inner step-off pad;
- Additional secondary step-off pads, within the posted area, may be used as necessary to restrict the spread of contamination out of the immediate area; and
- The final or outer step-off pad shall be placed immediately outside and adjacent to the CA.

E. Personnel Contamination Control

RCT

1. Require protective clothing as specified in the Radiological Work Permit (RWP) or other written authorization for entry into areas where removable contamination exists at levels exceeding those specified in Attachment A [§835.1102(e)].

2. Individuals exiting CAs, HCAs, ARAs, Radiological Buffer Areas (RBA) established for contamination control shall be monitored, as appropriate, for the presence of surface contamination [§835.1102(d)].

F. Conduct of Radiological Work

RCT

1. When evaluating work in radiologically posted areas, determine whether it is conducted in a manner that minimizes the spread of contamination to adjacent surfaces, individuals in the area, and the workplace atmosphere. Discuss contamination control related process improvements with the worker, their supervisor, the RCT supervisor or other personnel as appropriate. Document deficiencies in a logbook or other means and follow up to help assure work process improvements.

2. Use the following controls and techniques, as appropriate, to minimize the spread of contamination:

   - Minimization of individuals and materials entering contaminated areas;
   - Use of proven work techniques to minimize contamination spread;
   - Use of stop-work authority to correct radiological problems before they escalate;
   - Work area surveys to detect, and decontamination to reduce, contamination spread; and
   - Priority repairs of leaks to minimize the spread of contamination.

3. When removing smears from a CA, HCA or ARA, place the smears in a sealed package before removing them from the area. The sealed package can be a Whirl-Pak, Ziplock baggie, or other sealed container.

4. Field-screen all smears from a CA, HCA or ARA for gross contamination. Do not remove smears from a CA, HCA or ARA if the field screening indicates contamination levels greater than 100 times the surface contamination values in Attachment A [I/CATS Actions 8867 and 9067].

5. **IF** smears can not be field-screened for gross contamination due to interfering radiation (i.e., smears collected in an area posted as both Radiation Area and HCA), **THEN**
   properly containerized smears may be moved to a posted Radiological Area or RBA designated by the RCT Supervisor for field screening.

6. **IF** there are special circumstances that require highly contaminated smears to be removed (i.e., smears with levels greater than 100 times the surface contamination values in Attachment A) from a CA, HCA or ARA, **THEN**
   do so only with the permission of the PHP. Highly contaminated smears must be handled with special precautions including double sealed packaging and a documented removable contamination survey of the package exterior [I/CATS Actions 8867 and 9067].

7. Use a RWP in accordance with BJC-EH-4030 for counting smears and/or air samples at field counting stations or laboratory counting areas [I/CATS Actions 8867 and 9067].

8. For field counting stations, collect and count a minimum of one smear of the sample preparation counter top area upon completion of counting activities. Document this smear result with the survey for which the smears were collected [I/CATS Actions 8867 and 9067].

9. For fixed counting laboratories, perform at least one routine contamination survey for each shift of operation.

10. Dispose of smears counted at a field counting station or laboratory counting area as radioactive waste. Do not leave contaminated smears unattended in a Radioactive Materials Area unless properly packaged and labeled according to EH-4517.

G. Release of Material and Equipment from Radiological Areas

RCT

1. Identify the radionuclides that may be present in order to define the appropriate surface contamination release limits. Consult supervisor for guidance if needed.

2. Except as provided in Step G.4 and G.5, material and equipment in ~~CCZs,~~ RBAs established to control contamination, Soil Contamination Areas, CAs, HCAs, or ARAs shall not be released to a Controlled Area if [§835.1101(a)]:

   - Removable surface contamination levels on accessible surfaces exceed the removable surface contamination values specified in Attachment A [§835.1101(a)(1)]; **OR**
   - Prior use suggests that the removable surface contamination levels on inaccessible surfaces are likely to exceed the removable surface contamination values specified in Attachment A [§835.1101(a)(2)]; **OR**
   - There are inaccessible areas and the history of the item is unknown.

3. When performing surveys include a sufficient number of survey points to characterize the radiological condition of the surface of the item.

4. Material and equipment exceeding the removable surface contamination values specified in Attachment A may be conditionally released for movement on-site from one radiological area for immediate placement in another equally posted radiological area only if these items are [§835.1101(b)]:

   - Properly labeled/tagged according to the requirements of EH-4517;
   - Properly packaged to prevent the spread of contamination; and
   - Monitored and controlled by a trained radiological worker to ensure no unmonitored individual is likely to exceed a dose equivalent that would require monitoring in accordance with EH-4511 and EH-4512.

5. Material and equipment with fixed contamination levels that exceed the total contamination values specified in Attachment A may be released for use in controlled areas outside of radiological areas provided [§835.1101(c)]:

   - Removable contamination levels are below the values specified in Attachment A [§835.1101(c)(1)];
   - Materials are routinely monitored, clearly labeled/tagged to alert personnel of the contaminated status [§835.1101(c)(2)]; and
   - Controls are established to ensure no unmonitored individual is likely to exceed a dose equivalent that would require monitoring in accordance with EH-4511 and EH-4512.

H. Release of Material and Equipment to Uncontrolled Areas

NOTE: It is not the intent of Survey and Release Plans to substitute for Sampling and Analysis Plans or to be used to support release/disposal decisions based on volumetric contamination. Survey and Release Plans are used when surface contamination values in Attachment A apply; guidance for developing and implementing Survey and Release Plans can be found in Chapter 6 of EH-4500.

Project Health Physicist (PHP)

1. It is BJC policy that the release of large quantities (e.g., greater than approximately 30 m^3) of equipment and material should be performed with a written Survey and Release Plan in lieu of a 100% unconditional release survey; this includes equipment and materials to be released as a part of recycling or reuse efforts or to allow for the disposal in a DOE controlled or off-site industrial or sanitary landfill. Routine surveys for releasing small quantities of material and employee or subcontractor tools and equipment do not require a Survey and Release Plan.

NOTE: When a Survey and Release Plan is required, an independent evaluation by the Performance/Quality Assurance Organization is required in accordance with Directive 08.

RCT

2. Identify the radionuclides that may be present in order to define the appropriate surface contamination release limits. Consult supervisor for guidance if needed.

3. The criteria described in DOE Order 5400.5, *Radiation Protection of the Public and the Environment*, shall be followed for releasing material to uncontrolled areas and for releasing radioactive material contaminated in depth or volume. Examples of material potentially contaminated in volume include soil, wood, concrete, or other porous material. Materials that may be activated are also potentially contaminated in volume.

NOTE: The 80% administrative control limit only applies to surveys performed for the release of materials and equipment to uncontrolled areas.

4. Prior to being released, material and equipment that are potentially contaminated with radioactive material shall be surveyed to determine that both removable and total surface contamination levels (including contamination present on or under any coating) are less than 80% of the Attachment A values with the exception of the values listed in row 2 of Attachment A and are as low as reasonably achievable. The use of this 80% criterion is an administrative control limit. If an item has contamination greater than 80% of the Attachment A values but less than 100% of those values, only the PHP may allow release of the item if it is not reasonably achievable to decontaminate the item to less than the administrative control limit. Contamination from the radionuclides listed in row 2 of Attachment A must be less than 100% of the values. The typical minimum detectable activity for common radiological survey instruments exceeds 80 dpm/100 cm^2, but is less than the total contamination limit (for row 2 values of Attachment A) of 100 dpm/100 cm^2.

PHP

5. Custodial justification may be used to help the PHP determine if equipment and material may be potentially contaminated. Custodial justification, however, does not relieve the PHP from accountability for assuring the material or equipment meets the release limits as specified above.

NOTE: Custodial justification requires an equipment or material owner to certify by signature that equipment or material could not possibly be contaminated.

RCT

6. Any equipment or system component removed from a process that may have contacted radioactive material shall be considered contaminated until disassembled to the extent required using good judgement to perform an adequate survey, surveyed, and shown to meet the release limits as specified above. (For example, the ends of a pipe can be checked to determine the probability that the entire pipe interior is contaminated.)

7. Items with inaccessible surfaces that have been or suspected to have been in contaminated areas, or have the potential to be contaminated at levels greater than the release limits specified in Step H.5 of this procedure shall be treated as potentially contaminated. These items shall be subject to administrative controls unless the items are dismantled and monitored, or special survey techniques are used to survey all surfaces.

PHP

8. Evaluate wood, concrete, and other porous materials for release on a case by case basis.

RCT

9. Remove radiological labels/tags before release of material for unrestricted use.

10. Control material and equipment not immediately removed from CAs, HCAs, or ARAs to prevent contamination while awaiting release.

I. Documenting Contamination Surveys

RCT

1. Document contamination surveys on the contamination survey form, Attachment B, of this procedure, or an equivalent electronic record keeping system. Other methods of survey documentation are acceptable only when approved by the PHP. A logbook entry is not an acceptable equivalent to Attachment B.

2. Document contamination surveys as soon as reasonable after the survey is completed, but no later than the end of the next shift worked, unless awaiting laboratory results. For jobs continuing on multiple shifts, job conditions should be documented on survey forms and available for turnover and for reference related to current job conditions.

3. Contamination survey documentation shall contain the following information:

   - Date on which the survey was performed;
   - Identity of the individual who performed the survey;
   - Survey instrument type and identification number;
   - Survey tag number, if applicable; and
   - Results of the survey.

4. Records for release of surveyed material and equipment shall describe the following:

   - Property (uniquely identified in detail to allow future positive identification);
   - Date on which the survey was performed;
   - Identity of the individual who performed the survey;
   - Type and identification of the survey instrument used; and
   - Results of the survey.

5. Contamination survey forms should contain sufficient graphical information (i.e., location identifiers or maps) as appropriate to identify where contamination was detected.

NOTE: The five (5) working day review and approval deadline is reset when surveys are returned to the RCT because they are incomplete, inaccurate or illegible.

RCT Supervisor or designee

6. Review and approve the survey record no later than five (5) working days from the date of the survey to ensure it is complete, accurate, and legible.

RECORDS

Applicable records generated or received as a result of performing this procedure must be controlled.

The following records are generated by this procedure and SHALL be maintained in accordance with EH-4518, *Radiation Protection Program Records.*

1. Radiological Survey Forms
2. Survey and Release Plans
3. Area and Material/Equipment Release Surveys
4. Instrument Maintenance and Calibration Records

Record Requirements

SOURCE DOCUMENTS

- 10 CFR 835, *Occupational Radiation Protection*
- BJC/OR-301, *Bechtel Jacobs Radiation Protection Program for 10 CFR 835 Occupational Radiation Protection*
- BJC-OS-1001, *Records Management, Including Document Control*
- DOE G 441.1-9, *Radioactive Contamination Control Guide*
- DOE Order 5400.5, *Radiation Protection of the Public and the Environment*

Attachment A
SURFACE CONTAMINATION VALUES[1]
Page 1 of 1

Radionuclide	Removable [1,2,4]	Total (Fixed + Removable) [1,2,3]
U-nat, U-235, U-238, and associated decay products	[7]1,000	[7]5,000
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	20	100[8]/500
Th-nat, Th-232, Sr-90, Ra-233, Ra-224, U-232, I-126, I-131, I-133	200	1,000
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above[5]	1,000	5,000
Tritium and tritiated compounds[6]	10,000	N/A

1. The values in this attachment, with the exception noted in footnote 5, apply to radioactive contamination deposited on, but not incorporated into the interior or matrix of, the contaminated item. Where surface contamination by both alpha- and beta-gamma-emitting nuclides exist, the limits established for alpha- and beta-gamma-emitting nuclides apply independently.
2. As used in this table, dpm (disintegrations per minute) means the rate of emission by radioactive material as determined by correcting the counts per minute observed by an appropriate detector for background, efficiency, and geometric factors associated with the instrumentation.
3. The levels may be averaged over one square meter provided the maximum surface activity in any area of 100 cm^2 is less than three times the value specified. For purposes of averaging, any square meter of surface shall be considered to be above the surface contamination value if: (1) From measurements of a representative number of sections it is determined that the average contamination level exceeds the applicable value; or (2) it is determined that the sum of the activity of all isolated spots or particles in any 100 cm^2 area exceeds three times the applicable value.
4. The amount of removable radioactive material per 100 cm^2 of surface area should be determined by swiping the area with dry filter or soft absorbent paper, applying moderate pressure, and then assessing the amount of radioactive material on the swipe with an appropriate instrument of known efficiency. (Note—The use of dry material may not be appropriate for tritium.) When removable contamination on objects of surface area less than 100 cm^2 is determined, the activity per unit area shall be based on the actual area and the entire surface shall be wiped. It is not necessary to use swiping techniques to measure removable contamination levels if direct scan surveys indicated that the total residual surface contamination levels are within the limits for removable contamination.
5. This category of radionuclides includes mixed fission products, including the Sr-90 which is present in them. It does not apply to Sr-90 which has been separated from the other fission products or mixtures where Sr-90 has been enriched.
6. Tritium contamination may diffuse into the volume or matrix of materials. Evaluation of surface contamination shall consider the extent to which such contamination may migrate to the surface in order to ensure the surface contamination value provided in this appendix is not exceeded. Once this contamination migrates to the surface, it may be removable, not fixed; therefore, a "Total" value does not apply.
7. (alpha)
8. This value (100) is adopted from USNRC Regulatory Guide 1.86, "Termination of Operating Licenses for Nuclear Reactors," June 1974 and shall be used when releasing materials for unrestricted use.

Attachment B
EXAMPLE CONTAMINATION SURVEY FORM
Page 1 of 3

This attachment contains the actual form, beginning on the next page, which may be used directly.

Radiological Survey Form

Survey No.:_____ Date: _____ Time:_____ RWP NO:_____

Location of Survey – General (Site/Bldg.):_____ Specific (Room/Area/Item): _____

Purpose of Survey:_____

Remarks:_____

Technician:_____ Badge:_____ Technician:_____ Badge:_____

α Model #: Ludlum 2221 Serial #:_____ Cal. due date:_____ Probe Model #: Ludlum 43-5 Serial #:_____ Cal. due date:_____ Bkgd:_____ Ct. Time:_____ CF Pt.:_____ CF Plane:_____

βγ Model #: Ludlum 2221 Serial #:_____ Cal. due date:_____ Probe Model #: Ludlum 44-9 Serial #:_____ Cal. due date:_____ Bkgd:_____ Ct. Time:_____ CF Pt.:_____ CF Plane:_____

α Model #:_____ Serial #:_____ Cal. due date:_____ Probe Model #:_____ Serial #:_____ Cal. due date:_____ Bkgd:_____ Ct. Time:_____ CF Pt.:_____ CF Plane:_____

βγ Model #:_____ Serial #:_____ Cal. due date:_____ Probe Model #:_____ Serial #:_____ Cal. due date:_____ Bkgd:_____ Ct. Time:_____ CF Pt.:_____ CF Plane:_____

___Model #:_____ Serial #:_____ Cal. due date:_____ Probe Model #:_____ Serial #:_____ Cal. due date:_____ Bkgd:_____ CF:_____

Scaler Type:_____ Serial #:_____ Cal. due date:_____ Probe Model #:_____ Serial #:_____ Cal. due date:_____ α Bkgd:_____ Bkgd Ct. Time:_____ Ct. Time:_____ α CF:_____

βγ Bkgd:_____ Bkgd Ct. Time:_____ Ct. Time:_____ βγ CF:_____

Item No.	Removable α dpm/100cm^2	Total α dpm/100cm^2	Removable β,γ dpm/100cm^2	Total β,γ dpm/100cm^2	Sample Location and/or Remarks
Lc					N/A

Sketch of area/item surveyed
(Dose rates are underlined, smears are circled)

Released to:_____ RCT Supervisor Review:_____ / _____

signature / date

Note: Any response of the instrument that is above the Critical Detection Level (or Lc) is considered to be above background.

RADIOLOGICAL SURVEY CONTINUATION

Page ___ of

BUILDING:_____ LOCATION:_____ PURPOSE OF SURVEY:_____ SURVEY NO.:_____ RWP NO.:

TECHNICIAN:_____/_____BADGE NO:_____TECHNICIAN:_____/_____BADGE NO:

ITEM NO	TOTAL α dpm/ 100 cm²	LOOSE α dpm/ 100 cm²	TOTAL βγ dpm/ 100 cm²	LOOSE βγ dpm/ 100 cm²	SAMPLE LOCATION AND/OR REMARKS	ITEM NO	TOTAL α dpm/ 100 cm²	LOOSE α dpm/ 100 cm²	TOTAL βγ dpm/ 100 cm²	LOOSE βγ dpm/ 100 cm²	SAMPLE LOCATION AND/OR REMARKS

Source Documents and Other Documents Needed (Procedure Matrix)

Document Number	Document Title	Reference Number	Reference Title	Procedure	Regulation, Order, Standard, or Guide	Form	Other
EH-4516	Radioactive Contamination Control and Monitoring	BJC-EH-4006	Embryo/Fetus Protection	x			
EH-4516	Radioactive Contamination Control and Monitoring	BJC-EH-4030	Radiological Area Entry Control	x			
EH-4516	Radioactive Contamination Control and Monitoring	BJC-EH-4035	Radiation Safety Training	x			
EH-4516	Radioactive Contamination Control and Monitoring	BJC-GM-520	Documenting Radiation/Contamination Surveys	x			
EH-4516	Radioactive Contamination Control and Monitoring	Directive 08	PQA Review of Unconditional Release Surveys		x		
EH-4516	Radioactive Contamination Control and Monitoring	EH-4500	Radiological Control Operations Guide for the Environment Management and Enrichment Facilities at Oak Ridge, Tn, Portsmouth, OH, and Paducah, Ky	x			
EH-4516	Radioactive Contamination Control and Monitoring	EH-4511	External Dosimetry	x			
EH-4516	Radioactive Contamination Control and Monitoring	EH-4512	Internal Dosimetry	x			
EH-4516	Radioactive Contamination Control and Monitoring	EH-4513	Workplace Monitoring	x			
EH-4516	Radioactive Contamination Control and Monitoring	EH-4517	Posting and Labeling	x			
EH-4516	Radioactive Contamination Control and Monitoring	EH-4518	Radiation Protection Program Records	x			
EH-4516	Radioactive Contamination Control and Monitoring	SH-B-GLOS	Radiation Protection Glossary	x			
EH-4516	Radioactive Contamination Control and Monitoring	10 CFR 835	Title 10, Code of Federal Regulations, Part 835, (10 CFR 835) Occupational Radiation Protection.		x		
EH-4516	Radioactive Contamination Control and Monitoring	BJC/OR-301	Radiation Protection Program	x			
EH-4516	Radioactive Contamination Control and Monitoring	BJC-OS-1001	Records Management, including Document Control	x			
EH-4516	Radioactive Contamination Control and Monitoring	DOE G 441.1-9	Radioactive Contamination Control Guide		x		
EH-4516	Radioactive Contamination Control and Monitoring	DOE O 5400.5	Radiation Protection of the Public and the Environment		x		

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date April 24, 2006

PROCEDURE TO BE TRANSITIONED:

EH-4527

Functional
Procedure Type
(Company, Functional, Project, Subcontractor)

Receipt, Transport, and Movement of Radioactive Materials
(Procedure Number and Title)

January 12, 2006
(Procedure Date)

9
(Rev. Number)

Reason for Changes: Transition BJC document to PRS interim document

SECTION	DESCRIPTION OF CHANGE
Entire Document	Change "Bechtel Jacobs Company LLC" to "Paducah Remediation Services LLC"
Entire Document	Change "BJC" to "PRS", except in procedure numbers/titles
Entire Document	Replace BJC positions/titles with PRS positions/titles as indicated on attached matrix/crosswalk

Initiator/Originator: William R. Chase (Print Name / Sign Name) — 04/05/06 Date

REVIEWED BY: Neil C. Kielly (Discipline / Task Manager) — 4-6-06 Date

APPROVED BY: CARLOTAN EDMUNDS (Project Manager) — 4/08/06 Date

BJC to PRS Position/Title Matrix

BJC Positions	PRS Position
Alternate Source Custodian	Technical Program Specialist
BJC General Manager	PRS General Manager
BJC President	PRS President
Control Technicians	Radiation Control Technician or Dosimetry Technician
Deputy General Manager	Manager of Projects
Dosimetrist	Technical Program Specialist
Dosimetry Program Specialist	Technical Program Specialist
Dosimetry Specialist	Technical Program Specialist
ES&H Manager	ES&H Manager
ESH&Q Assessment Coordinator	ESH&Q Manager
Facility Manager	Facility Manager
Female General Employee	Female General Employee
Field Engineer	Field Engineer
Field Operations Manager	Field Operations Manager
Field Radiological Engineer	Radiological Engineer
First Line Manager	Supervisor
Functional Manager	Department Manager (Ex. RPPM for RADCON)
Health Physicist	Radiological Engineer
Instrumentation Supervisor	Technical Program Specialist for RADCON
Junior Radiological Control Technician	Junior Radiological Control Technician
Manager of Projects	Manager of Projects
On-scene Commander (Emergency Squad)	EOC Director
Operations Support Manager	Project Manager
Plant Shift Superintendent (USEC)	Plant Shift Superintendent (USEC)
Project Health Physicist	Radiation Protection Program Manager or designee
Project Line Management	Project Line Management
Project Manger	Project Manger
Project RADCON Manager	Radiation Protection Program Manager
Purchasing Agent	Subcontract Administrator
RADCON Internal Audit Coordinator (RAIC)	Technical Program Specialist
RADCON Manager	Radiation Protection Program Manager
RADCON Subcontractor	RADCON
RADCON Technician	Radiation Control Technician
Radiation Control Organization	RADCON
Radiation Control Technician	Radiation Control Technician
Radiation Control Technician Supervisor	Radiation Control Technician Supervisor
Radiation-generating Device Facility Manager	Facility Manager
Radiation-generating Device Operator	Radiation-generating Device Operator
Radiographers	Radiographers
Radiological Control Manager	Radiation Protection Program Manager
Radiological Control Officer	Radiation Protection Program Manager
Radiological Engineer	Radiological Engineer
Radiological Worker	Radiological Worker
Receiving Agent	Receiving Person
RWP User	RWP User
Safety Advocate	ESH&Q Safety Specialist
Senior Radiological Control Technician	Senior Radiological Control Technician
Site Dosimetry Coordinator	Technical Program Specialist
Site Dosimetry Manager	Technical Program Specialist
Source Custodian	Technical Program Specialist
Subcontract Coordinator	Subcontract Administrator
Subcontract Technical Representative	Project Manager
Subcontractor Assigned ES&H Representative	Assigned ES&H Representative
Supervisor	Supervisor
Supervisor of Declared Pregnant Worker	Supervisor of Declared Pregnant Worker
Technical Support Manager	Technical Program Specialist for RADCON
Training Manager	Training Manager
Work Group Supervisor	Supervisor

BECHTEL JACOBS
Bechtel Jacobs Company LLC

OWNER: Radiological Controls	**EH-4527**	**REV. NO. 9**
SUBJECT MATTER AREA: Transport; Survey	**PREPARER: Steve Green**	**Page 1 of 14**
DOC TYPE: Procedure	**CONCURRENCE/DATE: Steve Green 1/9/06 [Approval Signature On File]**	
If Doc Type is Procedure, identify what type: Technical ☐ Emergency ☐ Alarm Response ☐ Administrative ☒		
TITLE: RECEIPT, TRANSPORT, AND MOVEMENT OF RADIOACTIVE MATERIALS	**APPROVED BY/DATE: Steve Green 1/9/06 [Approval Signature On File]**	
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Exhibit L Mandatory Contractor Procedure? ☐	**If an Interim Procedure, Expiration Date:**	

CAUTION

This procedure contains commitments specific to Title 10, Code of Federal Regulations, Part 835, *Occupational Radiation Protection,* and the Bechtel Jacobs Company LLC (BJC) Issues and Corrective Action Tracking System (I/CATS). The source of the specific commitment follows the procedural step in brackets (e.g., [§835.405(b)] or [I/CATS Action ID 7511]).

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REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Intent change. Responsibilities shifted from BJC to RADCON subcontractor. Added section on receiving large shipping containers. Replaces SH-B-4025, Rev. 0, *Receipt, Transport and Movement of Radioactive Materials.*	2, 3, 4, 5, 6, 7, 9
1	Intent change. Added requirement for RCTs to review shipping papers. Deleted example of item to surveys from B.1. Deleted requirement to survey at 30 centimeters containers for shipping (I0057325). Provided clarification for surveying shipments	3, 5, 6, 7
2	Non-Intent Change. Changed note before step A.1.	2
3	Intent change. Major rewrite. Provided clarification and additional requirements for surveying and documenting shipments on DOE conveyances (I0058365). Revised survey form.	All
4	Intent Change. Combined Sections B and C. Deleted requirement for total contamination survey on packages to be shipped.	All
5	Non-intent Change. Clarified step B.9.	6
6	Intent Change. Deleted references to Site RADCON Manager, added references to Field Radiological Engineer.	3-9, 11
7	Administrative Change. Include reference to new form BJCF-919 and delete Attachment C.	3
8	Intent Change. Deleted unregulated requirements. Clarified text.	3 – 9
9	Administrative Change. Change the performer in Section F.3 to "RCT Supervisor or designee"	8

PURPOSE

This procedure provides instructions for performing radiological surveys on packages, materials, and conveyances used in the receipt and transportation of radioactive materials.

SCOPE

This procedure applies to Radiological Control (RADCON) personnel and those individuals qualified to conduct radiological surveys, primarily RADCON Technicians (RCT) and RCT Supervisors who review and approve radiological survey reports.

This procedure does not include all necessary survey provisions that apply to the preparation of shipments to Nevada Test Site.

OTHER DOCUMENTS NEEDED

- EH-4515, *Radiation Surveys*
- EH-4516, *Radioactive Contamination Control and Monitoring*
- EH-4517, *Posting and Labeling*
- EH-4518, *Radiation Protection Program Records*
- EH-4541, *Radiological Surveys to Support Waste Shipments to the Nevada Test Sit*
- SH-B-GLOS, *Radiation Protection Glossary*
- BJC-OS-1001, *Records Management, Including Document Control*
- Title 10, Code of Federal Regulations, Part 835, *Occupational Radiation Protection*
- Title10, Code of Federal Regulations, Part 71, *Packaging and Transportation of Radioactive Materials*
- Title 49, Code of Federal Regulations, Part 172, *Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements*
- Title 49, Code of Federal Regulations, Part 173, *Shippers – General Requirements for Shipments and Packagings*
- BJCF-919, Radiation/Contamination Survey – Transportation

WHAT TO DO

A. General Requirements

RCT

1. Use U.S. Department of Energy (DOE) surface contamination limits (i.e., 10 CFR 835, Appendix D), which are more limiting than U.S. Department of Transportation (DOT) (i.e., 49 CFR 173.443, Table 11) surface contamination limits for on-site movement of radioactive material and off-site radioactive material shipments.

2. Use DOT surface contamination limits for incoming radioactive material shipments on a non-DOE conveyance from an off-site origination and all subsequent on-site transfers up to the point where the receipt survey is performed (in accordance with §835.405).

3. Document all surveys required by this procedure using BJCF-919 or the site approved method; ensure all information required by this procedure is included in the survey.

B. Surveys for Shipments Received

NOTE: Surveys performed for receipt of radioactive material are performed to ensure compliance with 10 CFR 835.

RCT

1. Upon receipt of radioactive material, survey external surfaces of packages known to contain any of the following [§835.405(b)]:

a. Packages labeled with a Radioactive White I, Yellow II, or Yellow III label (as specified at 49 CFR 172.403 and 172.436-440) [§835.405(b)(1)];

NOTE: There are two definitions of exclusive use, which are provided in Attachment A. Any questions as to which definition to use should be directed to the Transportation Specialist.

b. Packages transported as low specific activity (LSA) material (as defined at 10 CFR 71.4) on an exclusive use vehicle (as defined at 10 CFR 71.4) [§835.405(b)(2)];

c. Packages that have evidence of degradation, such as packages that are crushed, wet, or damaged [§835.405(b)(3)].

2. **IF** monitoring is required by B.1 above, **THEN**
perform removable contamination surveys of packages known to contain radioactive material, unless the package contains only special form (as defined in 10 CFR 71.4) or gaseous radioactive material [§835.405(c)(1)].

Removable contamination measurements shall include at least one large area wipe [LAW] or smear on each package (more smears may be necessary to adequately characterize contamination on large packages). **IF** the LAW does not contain detectable contamination greater than the critical level (Lc), **THEN**
smears are not required provided Project Health Physicist (PHP) approval is obtained and provided that the removable surface contamination levels from row 2 or 3 in EH-4516, Attachment A do not apply.

3. **IF** removable contamination levels exceed the removable surface contamination levels in EH-4516, Attachment A, **THEN**

a. Properly control the package to prevent the spread of contamination; post the area, as appropriate, in accordance with EH-4517, *Posting and Labeling*.

b. Check the driver of the vehicle for contamination.

c. Check the vehicle for removable contamination.

d. Notify the RCT Supervisor.

RCT Supervisor

e. Notify the PHP, Field Radiological Engineer (FRE) and the Transportation Specialist.

RCT

4. **IF** monitoring is required by B.1 above, **THEN**
perform radiation surveys on packages known to contain radioactive material that emit significant beta, gamma, and neutron radiation, as applicable, unless the package contains less than a Type A quantity of radioactive material [§835.405(c)(2)]. Record the highest radiation measured at a distance of 30 cm from each package.

IF neutron radiation is not expected or a known ratio of neutron to gamma dose is available to determine total dose

AND

IF the package shielding prevents beta radiation from being detected, **THEN**
only gamma radiation surveys are necessary.

5. **IF** the radiation measurements exceed levels that would require posting according to EH-4517, **THEN**

a. Ensure that the package or area is appropriately controlled and posted.

b. Notify the RCT supervisor.

RCT Supervisor

c. Notify the PHP, FRE and the Transportation Specialist.

RCT

6. Complete the required monitoring as soon as practicable following notification of the receipt of the package, but no later than 8 hours after the beginning of the working day following receipt of the package [§835.405(d)].

7. **IF** the unloaded conveyance is no longer a radioactive material shipment and will not be returned to a Bechtel Jacobs Company LLC (BJC)-operated facility, or may potentially be released to the public, **THEN**
survey the conveyance for unrestricted release in accordance with EH-4516. Any questions as to the disposition of the conveyance should be directed to the Transportation Specialist.

NOTE: Sufficient survey points for transferable and direct should be obtained from conveyance to adequately characterize. This survey should include areas where packages were staged on conveyance and other horizontal surfaces of trailer. If contamination is detected on either the packages or on the conveyance then the rigor of survey should be increased to determine if contamination had migrated from conveyance loading surfaces.

8. **IF** contamination levels exceed the surface contamination levels in EH-4516, Attachment A, **THEN**

a. Notify the RCT supervisor.

RCT Supervisor

b. Notify the PHP, FRE and the Transportation Specialist.

9. **IF** the unloaded conveyance will be returned as a radioactive material shipment, **THEN**
follow Sections D and F of this procedure.

C. Survey of Reusable Radioactive Material Containers Prior to Use

NOTE 1: The requirements for the survey of reusable containers prior to loading for shipment to the Nevada Test Site can be found in EH-4541, *Radiological Surveys to Support Waste Shipments to the Nevada Test Site.*

NOTE 2: For the purposes of this procedure, dump trucks are not considered reusable radioactive material containers.

RCT

1. Attempt to identify the origin of the container and review available process history.

2. **IF** the container is new (i.e., recently manufactured and has not been previously used) and there is supporting process history, **THEN**
surveys prior to loading are not required.

3. **IF** the container was authorized to be transported from the Controlled Area by the BJC RADCON Subcontractor, **THEN**
surveys prior to loading are not required.

4. **IF** the container was delivered from another DOE facility **AND** survey documentation is provided that demonstrates the exterior surface contamination levels in EH-4516, Attachment A, are satisfied, **THEN**
surveys prior to loading are not required.

5. **IF** presented with a container survey that represents a current unrestricted release survey from a Nuclear Regulatory Commission or Agreement State facility or licensee, **THEN**
surveys prior to loading are not required.

6. **IF** there is no survey documentation available on the container, **THEN**
perform a contamination survey that is sufficient to characterize the radiological conditions of the exterior of the container.

7. **IF** exterior surface contamination levels exceed the surface contamination levels in EH-4516, Attachment A, **THEN**

   a. Control and/or post the area in accordance with EH-4517, as necessary.

   b. Notify the RCT Supervisor.

RCT Supervisor

   c. Notify the PHP, FRE and the Transportation Specialist.

D. Surveys of Packages Prior to Off-Site Shipment

NOTE 1: DOE removable surface contamination limits (i.e., 10 CFR 835, Appendix D), are more limiting than DOT (49 CFR 173.443, Table 11) removable surface contamination limits and apply for off-site radioactive material shipments [I/CATS Action ID 6875].

NOTE 2: The requirements for the survey of packages prior to shipment to the Nevada Test Site can be found in EH-4541.

NOTE 3: For the purpose of this procedure, Sealands, Intermodal containers and dump trucks may be considered packages.

RCT

1. Visually inspect the packages to be shipped to ensure they are not damaged and that there is no sign of leakage. **IF** the package is damaged or shows signs of loose material or leakage, **THEN**

   a. Notify the RCT Supervisor.

RCT Supervisor

   b. Notify the PHP, FRE and/or the Transportation Specialist.

RCT

2. Obtain at least three 100 cm^2 smears of the package or smear the entire package if the total surface area of the package is <300 cm^2. **IF** removable contamination levels exceed the removable surface contamination levels in EH-4516, Attachment A, **THEN**

   a. Ensure that the package is properly handled and placed in an appropriately posted area and/or labeled and tagged in accordance with EH-4517.

   b. Notify the RCT Supervisor.

3. Survey the surface of the package for the highest contact exposure rate. Survey the surface of the package for the highest 1 meter reading if Yellow II or III label is required. Surveys for gamma radiation only are acceptable if neutron radiation is not expected and the package shielding prevents significant beta radiation from being emitted [I/CATS Action ID 6875]. Perform surveys as required in Attachment B.

RCT Supervisor or designee

4. Review, sign and date the completed survey(s).

5. Provide a copy of the reviewed package survey(s) to the Transportation Specialist.

E. Survey of Conveyances Prior to Loading

RCT

1. Attempt to identify the origin of the conveyance and review available process history.

2. **IF** the conveyance was authorized to be transported from the Controlled Area by the BJC RADCON Subcontractor or there is sufficient process history to confirm that surface contamination levels are likely to be less than the surface contamination levels in EH-4516, Attachment A, **THEN** surveys prior to loading are not required.

3. **IF** there is not sufficient process history available on the conveyance to confirm that surface contamination levels are likely to be less than the surface contamination levels in EH-4516, Attachment A, **THEN** perform a contamination survey that is sufficient to characterize the radiological conditions of the conveyance.

4. **IF** contamination levels exceed the surface contamination levels in EH-4516, Attachment A, **THEN**

   a. Control and/or post the area in accordance with EH-4517, as necessary.

   b. Notify the RCT Supervisor.

RCT Supervisor

   c. Notify the PHP, FRE and the Transportation Specialist.

F. Survey of Loaded Conveyance Prior to Off-Site Shipment

NOTE: In the case of a flat-bed style vehicle, the outer surfaces of the vehicle are considered to be the vertical planes projected by the outer edges of the vehicle.

RCT

1. Survey the loaded conveyance at the locations noted in Attachment B. **IF** the front vertical plane/surface of the loaded conveyance does not exceed 2 mrem/hr on contact, **THEN** it is not necessary to survey the cab of the conveyance. Simply document "< 2 mrem/hr" on the DOT form.

2. Provide completed survey to RCT Supervisor.

RCT Supervisor or designee

3. Review survey documentation.

4. Forward all survey results to the Transportation Specialist for inclusion with the shipping papers.

G. Survey of Unloaded Conveyances at EMWMF Prior to Return to Generator

1. A conveyance being returned to generator after offloading its waste shall be surveyed for removable contamination and documented in accordance with EH-4516.

2. Removable contamination measurements shall include at least one LAW or smear on each conveyance and/or package (more smears may be necessary to adequately characterize contamination on large packages). **IF** the LAW does not contain detectable contamination greater than the critical level (Lc), **THEN**

   smears are not required provided PHP approval is obtained and provided that the removable surface contamination levels from row 2 or 3 in EH-4516, Attachment A does not apply.

RECORDS

The following records are generated by this procedure:

RADCON Organization

- Radiation/Contamination Surveys (complete and approved)

Record Requirements
Applicable records generated or received as a result of performing this procedure must be submitted to the Records Management and Document Control Center for record retention and disposition in accordance with the following:

- BJCF-919, Radiation/Contamination Survey – Transportation

SOURCE DOCUMENTS

- International Air Transportation Association, Dangerous Goods Regulations

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 4

A_1 and A_2 Quantity Limits – The regulations use the special form radioactive material (A_1) and normal form radioactive material (A_2) values as points of reference for quantity limits for each radionuclide. Each radionuclide is assigned an A_1 and A_2 value. These two values (in curies) are the maximum activity of that radionuclide that may be transported in a TYPE A package. The A_1 value is the limit of activity for a particular special form radionuclide in a Type A package. The A_2 value is the limit for the amount of activity that can be transported in a Type A package if the material is not in special form, i.e., "normal form."

BJC – Bechtel Jacobs Company LLC

Bulk Transport Conveyance – A transport vehicle or large freight container constituting bulk packaging in which radioactive material is loaded with no intermediate specification form of containment.

Carrier – Any entity engaged in the transportation of radioactive material as a common, contract, for hire or private carrier, or as a freight forwarder; or the U.S. Postal Service; or as officers, agent, and employees of such carriers.

Consignee – The entity designated in the shipping papers to receive the shipment.

Consignor – The entity executing the shipping papers and named as such in the shipping papers. The consignor is in every case a shipper.

Conveyance – (1) Any transport vehicle or large freight container for transport by public highway or rail; (2) Any vessel or hold, compartment, or defined deck area of a vessel including any transport vehicle on board the vessel for transport by water; and (3) Any aircraft used for transport by air.

DOE – U.S. Department of Energy

DOE Conveyance – Any conveyance used by or for DOE in the transportation of DOE radioactive material.

DOT – U.S. Department of Transportation

Exclusive use – The two definitions below apply to this procedure. Definition (a) is referenced by §835.405(b)(3) for the purpose of establishing monitoring requirements for package receipt. Definition (b) is used for guidance in implementation of DOE Order 460.2, *Department Materials Transportation and Packaging Management*, and is a broader definition which would also include exclusive use as defined in Definition (a).

(a) Is the sole use by a single consignor of a conveyance for which all initial, intermediate, and final loading and unloading are carried out in accordance with the direction of the consignor or consignee? The consignor and the carrier must ensure that any loading or unloading is performed by personnel having radiological training and resources appropriate for safe handling of the consignment. The consignor must issue specific instructions, in writing, for maintenance of exclusive use shipment controls, and include them with the shipping paper information provided to the carrier by the consignor (10 CFR 71.4).

Attachment A
ACRONYMS/DEFINITIONS
Page 2 of 4

(b) A service whereby a transport vehicle is assigned for the sole use of a specific consignor, loaded only by that consignor, and unloaded by the consignor or consignee. The carrier does not load any other consignor's freight in the vehicle and expedites delivery of the vehicle to the consignee (DOE G 460.2-1).

FRE – Field Radiological Engineer

Low Specific Activity (LSA) – All LSA materials have a characteristic of presenting limited radiation hazard, because of their relatively low concentration of radioactivity. The specific activity, in units of Curie per gram (Ci/g), are generally lowest in LSA-I and highest in LSA-III. When the specific activity of an LSA material is computed, the radioactivity is divided by the mass of material in which the radioactivity is distributed; the mass of the packaging that may surround the LSA is excluded from the calculation. The detailed description of the characteristics and limits for LSA-I, LSA-II, and LSA-III are indicated in §173.403 and 10 CFR 71.4, and they are unique for each LSA category.

LSA-I:

- Uranium and thorium ores, concentrates of uranium and thorium ores, and other ores containing naturally occurring radionuclides which are intended to be processed for the use of these radionuclides; or
- solid unirradiated natural uranium or depleted uranium or natural thorium or their solid or liquid compounds or mixtures; or
- radioactive material, other than fissile material, for which the A_2 value is unlimited; or
- Other radioactive material, excluding fissile material in quantities not excepted under § 173.453, in which the activity is distributed throughout and the estimated average specific activity does not exceed 30 times the values for activity concentration specified in § 173.436, or 30 times the default values listed in Table 8 of § 173.433.

LSA-II:

- water with tritium concentration up to 20.0 Ci/liter; or
- material in which the radioactive material is distributed throughout and the average specific activity does not exceed 10^{-4} A_2/g for solids and gases, and 10^{-5} A_2/g for liquids.

LSA-III:

- Solids, e.g., consolidated wastes and activated materials, that meet the leach test requirements of §173.468 and for which:
  - (i) the radioactive material is distributed throughout a solid or a collection of solid objects, or is essentially uniformly distributed in a solid compact binding agent, such as, concrete, bitumen, ceramic, etc.; and
  - (ii) the radioactive material is relatively insoluble, or it is intrinsically contained in a relatively insoluble material, so that, even under loss of packaging, the loss of radioactive material per package by leaching when placed in water for seven days would not exceed 0.1 A_2; and
  - (iii) the average specific activity of the solid does not exceed 2×10^{-3} A_2/g.

Non-DOE Conveyance – Any conveyance not used by or for DOE in the transportation of DOE radioactive material.

Attachment A
ACRONYMS/DEFINITIONS
Page 3 of 4

Normal Form Radioactive Material (A_2) – As defined in §173.403, normal form radioactive material means a Class 7 material that does not qualify as a "special form Class 7 material." Illustrated in Figure 3 are typical physical forms for normal form radioactive material.

Off-Site – Any area outside the boundaries of a DOE or contractor site and that portion of any DOE or contractor site to which the general public has unlimited access, including a public roadway.

On-Site – Any area within the boundaries of a DOE site to which access is continuously controlled.

Package – The packaging and its contents.

Packaging – The assembly of components necessary to ensure regulatory compliance. It may consist of one or more receptacles, absorbent materials, spacing structures, thermal insulation, radiation shielding, and devices for cooling or absorbing mechanical shocks. The conveyance, tie-down system, and auxiliary equipment may sometimes be designated as part of the packaging.

PHP – Project Health Physicist

RADCON – Radiological Control

Radioactive Material – Radioactive material means any material containing radionuclides where both the activity concentration and the total activity in the consignment exceed the values specified in the table in § 173.436 or values derived according to the instructions in § 173.433.

Radioactive Material Transportation – The movement of radioactive material by aircraft, rail, vessel, or highway vehicle when such movement is subject to DOT regulations or DOE Orders that govern such movements. Radioactive material transportation does not include preparation of material or packagings for transportation, monitoring required by 10 CFR 835, storage of materials awaiting transportation, or application of markings and labels required for transportation.

RCT – Radiological Control Technician

Shipment – Any off-site transportation of materials and equipment subject to DOT regulations or DOE Orders that govern such transportation.

Shipper – The organization and/or entity who offers a shipment to a carrier for transportation.

Specific Activity – As defined in §173.403, the activity of the radionuclide per unit mass of that nuclide. The specific activity of a material in which the radionuclide is essentially uniformly distributed is the activity per unit mass of the material.

Special Form Radioactive Material (A_1) – As defined in §173.403, special form radioactive materials are limited to those materials which, if released from a package, would present a hazard due to direct external radiation only. Usually, due to the high physical integrity of a special form material, radioactive material contamination is not expected even under severe accident conditions. This high physical integrity is occasionally the result of inherent natural properties of the material, such as its being in nondispersible solid form. Most often, however, it is an acquired characteristic, resulting from being welded (encapsulated) into an extremely durable metal capsule.

Attachment A
ACRONYMS/DEFINITIONS
Page 4 of 4

Transport Vehicle – Any conveyance (motorized or towed vehicle, railcar, aircraft, boat, barge, or seagoing vessel) used for the transportation of radioactive material.

Transportation – Inbound and/or outbound shipment of radioactive material from one point to another using a transport vehicle.

Type A Quantity – A quantity of radioactive material, the aggregate radioactivity of which does not exceed the A_1 or A_2 values, as appropriate, in Table A-1 of Appendix A to 10 CFR Part 71 (Also Table 173.435 of 49 CFR 173). Type A packaging is used for transportation of Type A quantities of radioactive material.

Attachment B
DOT REQUIRED SURVEYS

SURVEY REQUIREMENTS FOR DOE AND NON-DOE CONVEYANCES [a]

Object	Required Surveys
Packages in non-exclusive use vehicles	Contamination survey (A total area of 300 cm^2 must be wiped) Dose rate at 1 meter Dose rate at package surfaces
Closed, exclusive use vehicles and enclosed packages	Contamination survey (A total area of 300 cm^2 must be wiped) Dose rate in drivers compartment and all normally occupied areas [b] Dose rate at 2 meters from all lateral surfaces of closed vehicle Dose rate at all sides, top and lower exterior surface of closed vehicle Dose rate at package surface
Open flatbed, exclusive use vehicle without an exterior access limiting enclosure about the packages, and packages	Contamination survey (A total area of 300 cm^2 must be wiped) Dose rate in drivers compartment and all normally occupied areas [b] Dose rate at 2 meters from vertical planes of outer lateral edges of vehicle Dose rate at vertical planes of outer lateral edges of vehicle Dose rate at upper surfaces of "load" and lower external surface of vehicle Dose rate at all surfaces of packages
Open flatbed, exclusive use vehicle with an exterior access limiting enclosure about the packages, and packages in the enclosure	Contamination survey (A total area of 300 cm^2 must be wiped) Dose rate in drivers compartment and all normally occupied areas [b] Dose rate at 2 meters from vertical planes of outer lateral edges of vehicle Dose rate at vertical planes of outer lateral edges of vehicle Dose rate at upper surfaces of enclosure and lower external surface of vehicle Dose rate at all surfaces of packages inside enclosure

[a] Contact Transportation Specialist for limits for fissile material shipments, carriage by aircraft, carriage by closed vehicles used solely for transportation of RAM by highway or rail, and rail car shipments.
[b] If the front vertical plane/surface of the loaded conveyance does not exceed 2 mrem/hr on contact, then it is not necessary to survey the cab of the conveyance. Simply document "< 2 mrem/hr on the DOT form.

PADUCAH Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Tracey Brindley	**PRS-ENR-0020**	**REV. NO. 0**
SUBJECT MATTER AREA: Environmental Restoration/Environmental Monitoring	**PREPARER: Portage Environmental for Craig Jones**	**Page 1 of 13**
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TITLE: **Direct Push Technology Sampling**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 4/04/07**	
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REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release.	All

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1.0 PURPOSE

To describe the methods for collecting subsurface soil samples using Direct Push Technology (DPT) methods.

2.0 SCOPE

2.1 Scope of Procedure

2.1.1 This procedure applies to all types of subsurface soil sampling obtained using DPT methods completed by Paducah Remediation Services (PRS) or its subcontractors for the U.S. Department of Energy (DOE) at or near the DOE-owned Paducah Gaseous Diffusion Plant (PGDP).

2.1.2 This procedure applies to soil samples collected at depths equal to or greater than one foot below ground surface and extends to penetration limits of the DPT system employed for the sampling activity. Depth constraints are dependent on many variables including:

- The type of driving system used (e.g., the power and mass of the equipment used).
- The diameter of the down-hole tool and drive rod or casing being advanced.
- The resistance of subsurface materials against tool advance (which is controlled by formation density, degree of consolidation, and grain size).
- The soil friction acting on the down-hole tools, drive rod, or casing.

2.1.3 Any changes to or deviation from this procedure must be approved by the Project Manager (PM) or designee, prior to initiation of subsurface soil sampling using DPT methodologies.

2.2 Environmental, Safety, and Health Precautions

2.2.1 PRS policy is to maintain an effective program to control personnel exposure to chemical, radiological, and physical stress consistent with established standards of the contractor integrated safety management system, the Occupational Safety and Health Administration, and all other pertinent site-specific, state, and federal standards.

2.2.2 All field personnel will be required to wear personal protective equipment (PPE), as specified in the project-specific Environment, Health and Safety Plan, radiation work permit, and the site-specific work packages.

3.0 PREQUISITES

Project Manager or designee

3.1 Training

3.1.1 Collection of subsurface soil samples shall be limited to those individuals who are knowledgeable in the use of DPT and have a full knowledge and understanding of project-specified field procedures applicable to the collection of subsurface soil samples using DPT methodology.

NOTE: Limitations of use for DPT will be identified in the site-specific work packages.

3.2 Required Reading

NOTE 1: The PRS blue-sheeted Bechtel Jacobs Company LLC (BJC) procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in brackets will become the reference procedures once these procedures are approved and implemented by PRS.

NOTE 2: The following items may be reviewed and documented through briefings or required reading.

3.2.1 Field personnel must be knowledgeable of the latest version of all field and equipment operating procedures included below before beginning any subsurface soil collection activities using DPT methodology. Field personnel must have read and understood each procedure prior to collection of soil samples using DPT.

- BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*]
- BJC-ES-2700, *Field Logbooks* [PRS-ENM-2700, *Logbooks and Data Forms*]
- BJC-ES-2701, *Large Equipment Decontamination* [PRS-FCD-2701, *Large Equipment Decontamination*]
- BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* [PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*]
- BJC-ES-2704, *Trip, Equipment and Field Blank Preparation* [PRS-ENM-2704, *Trip, Equipment and Field Blank Preparation*]
- BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* [PRS-ENM-2708, *Chain-of-Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*]
- PA-5003, *Quality Assured Data-Paducah* [PRS-ENM-5003, *Quality Assured Data*]

3.2.2 Field personnel must be familiar with any project-specific documents that may include Health and Safety Plan, Quality Assurance Project Plan, Site-specific Sampling and Analysis Plan (SAP), and Waste Management Plan prepared for the specific project. A copy of each shall be available for review by field personnel before the onset and through the duration of any field activities. Consult these documents as necessary to obtain specific information regarding decontamination, equipment and supplies, health and safety, sampling collection and identification, sample packing, etc.

4.0 PROCEDURE

Project Manager or designee

4.1 Approvals and Notifications

4.1.1 Make appropriate notifications for sampling activity to be conducted at PGDP and associated areas.

4.1.2 Obtain an excavation/penetration permit in accordance with BJC-FS-1004, *Excavation/Penetration Permit* [PRS-WCE-0026, *Excavation/Penetration Permit*] and any other required permits (i.e., radiological work permit).

4.2 Special Tools, Equipment, Parts and supplies

NOTE: A DPT system generally consists of a sampling tool [composed of a drive tool body, tip (for some tools), a sample liner or chamber], a drive rod or casing extending from the sampling zone to the surface, and the equipment used to advance the tool or logging device and rod or casing.

4.2.1 Ensure that the following items are available, as required, and ready for use prior to initiating any subsurface soil sampling in the field. Depending on the purpose of the sampling effort(s) as reflected in the field sampling plan, not all equipment may be needed.

- Site-specific plans
- Field logbook
- DPT equipment or Geoprobe® platform including tubes sleeves, split-spoon, pushrods, etc.
- Calibrated photo-ionization detector (PID) or other organic vapor analyzer.
- Camera
- Indelible black ink pens and markers
- Measuring tape
- Appropriate sample containers
- Deionized (DI) water
- Insulated cooler and waterproof sealing tape

- Labels and appropriate forms/documentation for sample shipment including chain-of-custody forms, custody seals, and sample labels
- Ice bags or “blue ice”
- Plastic zip-lock bags
- Personal protective clothing and equipment
- Stainless steel and/or Teflon-lined spatulas/pans/trays/bowls/wood spatulas
- Plastic sheeting
- Decontamination equipment and supplies
- Health and safety monitoring equipment
- Two-way radio or cellular telephone
- Appropriate containers for investigation-derived wastes as specified in the site-specific Waste Management Plan
- Sampling equipment shall include all associated supplies necessary to complete sampling such as stainless steel liners, Teflon tape, plastic liner caps, etc.
- Portable electronic balance.

Field Team Leader

4.3 Pre-Performance Activities

NOTE 1: A brief summary of subsurface soil collection methods using DPT methodologies is included in Attachment B, “Summary of Sample Collection Methods.”

NOTE 2: Inspect threads on drill rods for wear and make sure the Geoprobe® hammer is properly charged with nitrogen prior to equipment use.

4.3.1 Don the appropriate personal protective clothing as specified in the site-specific health and safety plan and radiation work permit.

4.3.2 Survey and stake sampling locations, as required, and determine appropriate sampling requirements.

4.3.3 Place clean plastic sheeting on a flat and level surface near the sampling area, if possible.

4.3.4 Place decontaminated equipment to be used on the plastic and place the sample cooler(s) on separate plastic sheeting.

4.3.5 Cover all equipment and supplies with clean plastic sheeting when not in use.

4.3.6 Set up the decontamination line required by the site-specific SAP. The decontamination line should be staged on plastic sheeting.

Field Team Members

4.4 Collection of Subsurface Soil Samples – General Steps

NOTE 1: Follow these general steps when collecting any subsurface soil samples using methods described in Sections 4.5 or 4.6.

NOTE 2: Maintain the proper orientation of the sample in a casing as noted in Attachment B (Sample Orientation). Multiple samples, representing different depths, can be obtained from a single retracted casing. Additionally, for volatile organic compound (VOC) investigations, a field PID can be used to pinpoint depth of contamination from a single extracted casing if biased sampling is called for in the SAP.

NOTE 3: Powered equipment may be used to acquire soil samples from any depth (surface, shallow subsurface and deep subsurface). When powered equipment is used to collect the sample, care must be taken that exhaust fumes, gasoline, and\or oil do not contaminate the borehole or the sample. To prevent cross contamination of samples, **DO NOT** re-fuel or service powered equipment within a 25 foot radius of the sample location.

4.4.1 **IF** sampling locations defined in the SAP, including sample depth, cannot be sampled due to a physical obstruction, **THEN** contact the PM or designee.

4.4.2 **ONCE** the project manager or designee approves a change, **THEN** move the sampling location **AND** document the change in the field logbook **AND** notify the data manager.

4.4.3 Document the sampling events by recording the information in the designated field logbook as defined in BJC-ES-2700, *Field Logbooks* [PRS-ENM-2700, *Logbooks and Data Forms*].

4.4.4 Document any and all deviations from any relevant procedures in the field logbook and include rationale for any procedural changes.

4.4.5 Follow applicable sampling requirements related to data quality as described in PA-5003 *Quality Assured Data-Paducah* [PRS-ENM-5003, *Quality Assured Data*].

4.4.6 Sample containers should be labeled prior to collection of the sample. Enter necessary information on the sample label [e.g., date and time (24-hour clock)] at the time of sample collection. See BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* [PRS-ENM-2708, *Chain-of-Custody forms, Field Sample Logs, Sample Labels, and Custody Seals*].

4.4.7 Complete chain of custody forms as described in BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* [PRS-ENM-2708, *Chain-of-Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*]

4.4.8 If applicable, collect quality control samples in accordance with

BJC-ES-2704, *Trip, Equipment and Field Blank Preparation* [PRS-ENM-2704, *Trip, Equipment and Field Blank Preparation*].

4.5 Collecting Samples for Volatile Organic Compound Analysis

NOTE: Follow these requirements for collecting grab samples of subsurface soil for volatile organic compounds (VOCs) or other samples degraded by aeration.

4.5.1 Soil samples collected for VOC analysis always will be collected first to minimize loss of volatiles due to disturbance and off gassing. Collect VOC samples with minimum amount of disturbance as possible.

NOTE: The selected method of collection may vary depending upon site conditions, data quality objectives, and the degree of known or suspected contaminant(s).

4.5.2 Collect VOC samples as undisturbed grab samples to minimize the potential loss of VOCs due to volatilization using

- Method or sampling device with Teflon or stainless steel sample liners, or other approved material **OR**
- Method and equipment specified in the project-specific SAP.

4.5.3 **IF** the sampling method itself does not already seal the material in a container, **THEN** place the soil in each of the designated containers.

4.5.4 Ensure that no headspace is left in the container and seal the container.

4.5.5 Complete any necessary health and safety field screening for volatiles and radionuclides using the instruments specified in the site-specific documents, including the radiation work permit.

4.6 Collecting Samples for Nonvolatile or inorganic Compound Analysis

NOTE: Follow these requirements for collecting samples of subsurface soil for nonvolatile organic or inorganic analyses.

4.6.1 Collect the required sample volume using the sampling device specified in the site-specific SAP.

4.6.2 Complete any necessary health and safety field screening for volatiles and radionuclides using the instruments specified in the site-specific documents, including the radiation work permit.

4.6.3 Transfer the soil collected directly into a clean stainless steel, Teflon-lined tray or bowl, or other project approved devices or material.

4.6.4 **IF** grab sample is being collected, **THEN** advance to 4.6.7.

4.6.5 **IF** composite samples are being collected, **THEN** transfer each aliquot (sub sample) directly into a clean stainless steel or Teflon lined troy or bowl.

4.6.6 Homogenize the sample, if applicable, by thoroughly mixing the total sample volume using a decontaminated stainless steel or Teflon-lined spoon, spatula, or trowel.

4.6.7 Use the spoon, spatula, or trowel to distribute the sample volume into the appropriate labeled sample containers (including the required quality control samples).

4.6.8 Secure the appropriate cap on each container immediately after filling.

4.7 Post performance Activities

4.7.1 Wipe sample containers clean with a clean Kimwipe or paper towel.

4.7.2 Record required information such as sample collection time and date on sample label, field custody form, logbook, and other data collection forms.

4.7.3 Place samples in a sample cooler containing ice bags or “blue ice” to maintain a preservation temperature of 4°C, as required. Ensure that the temperature of the samples is maintained in accordance with GEO-TEC-017, *Temperature Control for Sample Storage* [PRS-ENM-0021, *Temperature Control for Sample Storage*] until they are delivered to the laboratory.

4.7.4 If samples are to be shipped off-site, ship according to TR-9503, *Shipping Samples from a Bechtel Jacobs Company Site* [PRS-WSD-9503, *Off-Site Sample Shipping*].

4.7.5 Decontaminate equipment according to BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* [PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*] and BJC-ES-2701, *Large Equipment Decontamination* [PRS-FCD-2701, *Large Equipment Decontamination*].

4.7.6 Copy and submit field data forms (including all automatic data recording media, such as print-outs, logs, disks or tapes, and activity-specific data collection forms) as required by BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*].

4.7.7 Obtain radiation-screening results, as necessary, for shipments to an off-site laboratory.

4.7.8 Complete logbook in accordance with BJC-ES-2700, *Field Logbooks* [PRS-ENM-2700, *Logbooks and Data Forms*].

4.7.9 Monitor all samples for radioactivity before shipment is sent off-site to ensure total and transferable surface contamination for various radionuclides are below allowable levels. Department of Transportation off-site shipments may require dose rate survey of the package, per the direction of the Health Physicist.

4.7.10 Record Global Positioning System readings, if required, in the field log book, **AND** transfer the data into the Sample Management Office for inclusion in PGDP databases (e.g., PEMS).

4.7.11 Dispose of the excess sample media, personnel protective equipment, plastic, and other waste materials as specified by the Waste Management Plan, Statement of Work, or other applicable documents.

5.0 RECORDS

5.1 General Requirements

5.1.1 Records generated as a result of this procedure or reference procedure(s) shall be processed and maintained in accordance with requirements in BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*].

5.2 Quality Records

- Field logbooks
- Chain-of-custody forms

5.3 Non-Quality Records

- None

6.0 SOURCE DOCUMENTS

- McCall W., Nielsen D.M., Farrington S.P., and Christy T.M. "Use of Direct-Push Technologies in Environmental Site Characterization and Ground-Water Monitoring," *Practical Handbook of Environmental Site Characterization and Ground –Water Monitoring*, Second Edition. CRC press, 2006. p 345-471.
- U.S. Environmental Protection Agency (EPA), November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual,* Section 11. Region 4, Environmental Compliance Branch, Athens, GA.
- GEO Consultants, LLC, Surface Soil Sampling GEO-TEC-020, Revision 2, 2006.

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 1

DEFINITIONS

COMPOSITE SAMPLE - A sample collected at a given point in time consisting of a series of discrete samples (aliquots) that have been mixed together.

GRAB SAMPLE – An individual sample or aliquot collected from a single location at a specific time or period of time.

SPOON/SCOOP – A small stainless steel or Teflon-lined utensil approximately 6 to 8 in. in length with a stem-like handle.

SUBSURFACE SOIL – The soil that exists 1 ft (30 cm) or more below ground surface.

TROWEL – A small stainless steel shovel approximately 6 in. to 8 in. in length with a slight (~140º) curve and a stem-like handle.

WOOD SPATULA – A small wood strip approximately 5/8 in. by 8 in.

ACRONYMS

DI – deionized
DOE – U.S. Department of Energy
DPT – Direct Push Technology
PEMS – Project Environmental Measurement Systems
PGDP – Paducah Gaseous Diffusion Plant
PID – photo-ionization detector
PM – Project Manager
PPE – personal protective equipment
PRS – Paducah Remediation Services, LLC
SAP – Sampling and Analysis Plan
VOA – volatile organic analysis
VOC – volatile organic compound

Attachment B
SUMMARY OF SAMPLE COLLECTION METHODS

DPT technology includes a variety of methods for collecting samples of environmental media. Soil samples are obtained using DPT machines that advance a sampling tool by pushing (using hydraulic rams and the static weight of the machine), driving (using hydraulic or mechanical percussion hammers), or vibrating (using a vibratory drive head). The DPT machines typically used range from small, lightweight all-terrain or tracked vehicles to cargo vans and pickup trucks. The tools typically are advanced using either one string or two concentric strings of small-diameter rods or drive casing. Rotary drilling action generally is not used to advance the rods or tools and essentially no drill cuttings are generated from the DPT soil sampling process. Some small diameter sampling tools can even be advanced manually, using “slam bars” or hand-held percussion hammers.

Direct Push Rigs

This method uses a standard split-spoon modified with a locking tip which keeps the spoon closed during the sampling push. Upon arrival at the desired depth, the tip is remotely released and the push continued. During the push, the released tip moves freely inside of the spoon as the soil core displaces it. This technique is particularly beneficial at highly contaminated sites, because no cuttings are produced. The push rods generally are retrieved with very little residue resulting in minimal exposure to sampling personnel and reduced investigation-derived waste. The residue on the rods shall be handled as cuttings.

Before the soil is placed in a pan, it is necessary to remove the top several inches of soil to minimize the possibility of cross-contamination of the sample from fall-in of material from the upper portions of the hole. The soil removed due to fall in material shall be handled as cuttings. Once the soil is placed in a pan, it is thoroughly mixed, (Section 4.6.6), and placed in the appropriate sample container(s). Section 4.5 contains specific procedures for collecting and handling soil samples for volatile organic compounds analysis.

Geoprobe® Large Bore Soil Sampler

Geoprobe® is the brand name of widely used DPT equipment specifically designed for the environmental industry and offers several tools for soil sample collection. Among these are the Macro-Core® Soil Sampler and the large bore and dual tube soil sampling systems.

The Large Bore sampler is a solid barrel, piston sealed, direct push device for collecting discrete interval samples of unconsolidated materials at depth. The sampler is approximately 30-inches (762 mm) long and has a 1.5-inch (38 mm) outside diameter. The Large Bore sampler is capable of recovering a discrete sample core 22 inches x 1.0 inches (559 mm x 25 mm) contained inside a removable liner. Sample volume measures up to 283 ml. The liner is a 24-inch long by 1.15-inch outside diameter (610 mm x 29 mm) removable/replaceable thin-walled tube that fits inside the Large Bore sample tube. Liners facilitate retrieval of the sample and may be used for storage, when applicable. The Large Bore soil sampler is pushed with 1.25-inch diameter probe rod.

The following is a step-by-step description of the components and procedures used to collect a soil sample with the Large Bore sampler.

Attachment B
SUMMARY OF SAMPLE COLLECTION METHODS
Page 2 of 3

Assembly of Large Bore Sampler

1. Select a liner tube and push on to cutting shoe (one end of liner should be slightly flared, push this end on to shoe).

2. Insert end of tube opposite cutting shoe into sample tube and screw cutting shoe firmly into sample tube.

3. Thread piston tip onto piston rod, then run piston assembly through the end of the sample tube opposite the cutting shoe, seating the piston tip in the cutting shoe. There should be a short section of exposed piston rod sticking out of sample tube.

4. Place drive head over exposed end of piston rod and thread into end of sample tube.

5. Install piston stop-pin in top of drive head. This retains the piston rod assembly during the push.

6. The Large Bore sampler now is fully assembled and ready for sample collection.

Sample Collection Using Large Bore Sampler

1. Attach assembled Large Bore sampler to end of probe rod.

2. Attach drive cap to probe rod and push rod into ground.

3. Add additional rods to push Large Bore sampler to target sampling depth.

4. At the desired target sampling depth, remove the drive cap to access inside of probe rod. Couple extension rods and extension rod handle together and insert into probe rod. Using handle, turn the extension rods inside probe rod. This should engage the piston-stop pin and remove it from the drive head attached to the top of the sample tube. Retrieve extension rods and attached piston-stop pin. Add additional probe rod, if required; reattach drive cap to top of probe rod and push probe rod and Large Bore sampler 24 inches to fill sampler.

5. Attach pull cap and retrieve tool string.

6. When retrieved, remove the piston rod, with piston tip, and the drive head.

7. Using the Large Bore wrench, unscrew the cutting shoe from the down-hole end of the sample tube.

8. Remove the cutting shoe and attached liner and sample from sample tube.

9. Sample now has been collected and is contained in the liner. At this time, it can be sub-sampled per analytical requirements.

Attachment B
SUMMARY OF SAMPLE COLLECTION METHODS
Page 3 of 3

Liner Use and Material Selection

Due to the mode of operation, the Large Bore soil sampler must be used with a liner. Liners are available in the following materials: stainless steel, brass, cellulose acetate butyrate (CAB), and Teflon®. If samples are collected for organic compound analyses, Teflon® liners are required. CAB liners may be used if metals or other inorganic constituents are the object of the investigation.

Sample Orientation

When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It also is important to maintain proper orientation to define precisely the depth from which an aliquot was collected. Maintaining proper orientation typically is accomplished using vinyl end caps. Convention is to place red caps on the top of liner and black caps on the bottom to maintain the proper sample orientation. Orientation also can be indicated by marking on the exterior of the liner with a permanent marker.

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PADUCAH Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Tracey Brindley	**PRS-ENR-0021**	**REV. NO. 0**
SUBJECT MATTER AREA: Environmental Restoration/Environmental Monitoring	**PREPARER: Portage Environmental for Craig Jones**	**Page 1 of 9**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** Tracey Brindley (Signature on File in DCC) 3/14/07	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **Tree-Tissue (Tree Core) Sample Collection**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 4/04/07**	
USQD/UCD No: USQD-PH-SITE-0051/UCD-PH-SITE-0091	**REQUIRED REVIEW DATE: 4/04/10**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

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Revision Number	Description of Changes	Pages Affected
0	Initial Release.	All

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1.0 PURPOSE

To describe the method and equipment used to collect and package tree core samples for analytical analysis.

2.0 SCOPE

2.1 Scope of Procedure

2.1.1 This procedure applies to all tree core samples obtained for analytical analysis that are coordinated by Paducah Remediation Services (PRS) and subcontractors for the U.S. Department of Energy (DOE) at and near the DOE owned Paducah site.

2.1.2 Any changes to or deviation from this procedure must be approved by the Project Manager or designee prior to starting any tree core sample collection for analytical analysis.

2.2 Environment, Safety, and Health Precautions

2.2.1 PRS policy is to maintain an effective program to control personnel exposure to chemical, radiological, and physical stress consistent with established standards of the contractor integrated safety management system, the Occupational Safety and Health Administration, and all other pertinent site-specific, state, and federal standards.

2.2.2 All field personnel will be required to wear personal protective equipment (PPE) as specified in the project-specific environment, health and safety plan and the site-specific work packages.

3.0 PREQUISITES

Project Manager or designee

3.1 Training

3.1.1 Collection of tree core samples shall be limited to those individuals who are trained and knowledgeable of all field procedures applicable to the collection of tree core samples for analytical analysis.

3.2 Required Reading

NOTE 1: The PRS blue-sheeted Bechtel Jacobs Company LLC (BJC) procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in brackets will become the reference procedures once these procedures are approved and implemented by PRS.

NOTE 2: The following items may be reviewed and documented through briefings or required reading.

3.2.1 Field personnel must be knowledgeable of the latest version of all field and equipment operating procedures included below before obtaining any tree cores.

- BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*];
- BJC-ES-2700, *Field Logbooks*, [PRS-ENM-2700, *Logbooks and Data Forms*];
- BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* [PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*];
- BJC-ES-2704, *Trip, Equipment and Field Blank Preparation* [PRS-ENM-2704, *Trip, Equipment and Field Blank Preparation*];
- BJC-ES-2708, *Chain-of- Custody Protocol for Environmental Sampling* [PRS-ENM-2708, *Chain-of-Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*];
- PA-5003, *Quality Assured Data-Paducah* [PRS-ENM-5003, *Quality Assured Data*].

3.2.2 Field personnel must be familiar with any project-specific documents that may include health and safety plan, quality assurance project plan, site-specific sampling and analysis plan (SAP), and waste management plan prepared for the specific project. A copy of each shall be available for review by field personnel before the onset and through the duration of any field activities. Consult these documents as necessary to obtain specific information regarding decontamination, equipment and supplies, health and safety, sampling collection and identification, sample packing, etc.

4.0 PROCEDURE

Project Manager

4.1 Approvals and Notifications

4.1.1 Make appropriate notifications for sampling activity to be conducted at Paducah Gaseous Diffusion Plant (PGDP) and associated areas.

4.1.2 Obtain any required permits (i.e., excavation or penetration permit, radiological work permit).

4.2 Special Tools, Equipment, Parts, and supplies

4.2.1 Ensure that the following items are available, as required, and ready for use prior to initiating any subsurface soil sampling in the field. Depending on the purpose of the sampling effort(s) as reflected in the field sampling plan (FSP), not all equipment may be needed.

- Site-specific plans
- Field logbook
- A tree increment borer that includes a handle, an auger (approximately 4.3 millimeter inner diameter) and an extraction rod (spoon).
- Borer sharpening kit
- Borer lubricant (Silicon grease)
- Indelible black ink pens and markers
- Measuring tape
- Appropriate sample containers
- Deionized water
- Insulated cooler and waterproof sealing tape
- Labels and appropriate forms/documentation for sample shipment including chain-of-custody forms, custody seals, and sample labels.
- Ice bags or “blue ice”
- Plastic zip-lock bags
- Personal protective clothing and equipment
- Plastic sheeting
- Decontamination equipment and supplies
- Health and safety monitoring equipment
- Two-way radio or cellular telephone
- Appropriate containers for investigation-derived wastes as specified in the site-specific waste management plan

Field Team Members

4.3 Pre-Performance Activities

4.3.1 Don the appropriate personal protective clothing as specified in the site-specific health and safety plan. PPE should be worn for radiological areas in accordance with radiation work permit.

4.3.2 Survey and stake sampling locations, as required, and determine appropriate sampling requirements.

4.3.3 Place clean plastic sheeting on a flat and level surface near the sampling area, if possible.

4.3.4 Place decontaminated equipment to be used on the plastic and place the sample cooler(s) on separate plastic sheeting.

4.3.5 Cover all equipment and supplies with clean plastic sheeting when not in use.

4.3.6 Set up the decontamination line required by the site-specific SAP. The decontamination line should be staged on plastic sheeting.

Field Team Members

4.4 Collection of Tree Core Samples

Collecting tree tissue samples (tree core) is done using a tree increment

borer. This method is less intrusive and generates less investigation derived waste than cutting pieces from the tree with a saw. A tree increment borer is a tool that includes a handle, an auger and an extraction rod, called a spoon. The handle and the auger fit together to form a T-shaped hand drill. The auger is hollow, so that when it's drilled into a tree, a core of wood is forced into the interior of the tool. The wood core then can be removed by sliding the spoon between the core and the inner surface of the auger. Tree borers come in various lengths.

4.4.1 **IF** sampling locations defined in the SAP cannot be sampled due to a physical obstruction, **THEN** contact the PM.

4.4.2 **ONCE** the project manager approves a change, **THEN** move the sampling location **AND** document the change in the field logbook **AND** notify the data manager.

4.4.3 Document the sampling events by recording the information in the designated field logbook as defined in BJC-ES-2700, *Field Logbooks* [PRS-ENM-2700, *Logbooks and Data Forms*].

4.4.4 Document any and all deviations from any relevant procedures in the field logbook and include rationale for any procedural changes.

4.4.5 Follow applicable sampling requirements related to data quality as described in PA-5003 *Quality Assured Data-Paducah* [PRS-ENM-5003, *Quality Assured Data*].

4.4.6 Sample containers should be labeled prior to collection of the sample. Enter necessary information on the sample label [e.g., date and time (24-hour clock)] at the time of sample collection. See BJC-ES-2708, *Chain-of-Custody Protocol for Environmental Sampling* [PRS-ENM-2708, *Chain-of-Custody forms, Field Sample Logs, Sample Labels, and Custody Seals*].

4.4.7 Collect the required sample volume as specified in the SAP using the coring device as specified in Tree core extraction steps 1 through 6.

NOTE 1: For ease of use, always keep the auger drill sharp using the sharpening kit. Additionally, if the auger is extremely difficult to advance, some silicon grease may be used as lubrication. Use the silicon grease to lubricate the boring tool **only** if necessary.

NOTE 2: Numerous core samples may need to be taken to get appropriate volume.

Tree core extraction steps

1. If not otherwise specified in the FSP, find a flat surface on the tree at 4.5 feet (1.37 m) above the ground, which is in an accessible

location.

2. Remove the spoon from the auger and put it on a decontaminated surface somewhere you know you will find it, and remember not to step on it.

3. To ensure that the pith of the tree is intersected, position the auger on the bark so that the axis of the borer will pass through the center of the tree.

4. While holding firmly and pushing toward the center of the tree, turn the borer clockwise several times very slowly with one hand on the auger near the tree, and the other hand at the very top of the auger, until the threads penetrate solid wood for at least an inch, or until the auger will support itself during this initial stage of coring. Try to maintain the projection of the auger toward the center of the tree. Once the auger will support itself, continue turning the auger until it reaches the center of the tree or until it can no longer be turned.

5. Insert the spoon into the auger carefully, with the empty U-side up. Press gently but firmly until the spoon is completely inside the borer. Take care to not bend the spoon. Once the spoon is all the way in, turn the auger one (and only one) full turn counter-clockwise. This will break the core off inside the spoon.

6. Carefully and slowly remove the spoon. The core will come out balanced on top of the spoon. Be aware while removing the spoon, the core can easily fall and break. Once the core and spoon are completely removed from the auger, the bark should be carefully removed from the core.

4.4.8 Complete any necessary health and safety field screening for volatiles and radionuclides using the instruments specified in the site-specific health and safety plan.

4.4.9 Transfer the core(s) collected directly into the appropriate sample container as specified by the SAP.

4.4.10 Secure the appropriate cap on each container immediately after filling.

4.4.11 Complete chain of custody forms as described in BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* [PRS-ENM-2708, *Chain-of-Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*].

4.4.12 If applicable, collect quality control samples in accordance with BJC-ES-2704, *Trip, Equipment and Field Blank Preparation* [PRS-ENM-2704, *Trip, Equipment and Field Blank Preparation*].

4.4.13 **IF** not using a dedicated boring tool per sample, **THEN**

decontaminate tool according to BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* [PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*] to thoroughly clean the tool between sampling locations, unless otherwise specified.

4.5 Post Performance Activities

4.5.1 Wipe sample containers clean with a clean Kimwipe or paper towel.

4.5.2 Record required information such as sample collection time and date on sample label, field custody form, logbook, and other data collection forms.

4.5.3 Place samples in a sample cooler containing ice bags or “blue ice” to maintain a preservation temperature of 4°C, as required. Ensure that the temperature of the samples is maintained in accordance with GEO-TEC-017, *Temperature Control for Sample Storage* [PRS-ENM-0021, *Temperature Control for Sample Storage*] until they are delivered to the laboratory.

4.5.4 If samples are to be shipped off-site, ship according to TR-9503, *Shipping Samples from a Bechtel Jacobs Company Site* [PRS-WSD-9503, *Off-Site Sample Shipping*].

4.5.5 Decontaminate sampling equipment according to BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* [PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*].

4.5.6 Copy and submit field data forms (including all automatic data recording media, such as print-outs, logs, disks or tapes, and activity-specific data collection forms) as required by BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*].

4.5.7 Obtain radiation-screening results, as necessary, for shipments to an off-site laboratory.

4.5.8 Complete logbook in accordance with BJC-ES-2700, *Field Logbooks* [PRS-ENM-2700, *Logbooks and Data Forms*].

4.5.9 Monitor all samples for radioactivity before shipment is sent off-site to ensure total and transferable surface contamination for various radionuclides are below allowable levels.

4.5.10 Record global positioning system readings, if required, in the field log book, **AND** transfer the data into the Sample Management Office for inclusion in PGDP databases (e.g., PEMS).

4.5.11 Dispose of the excess sample media, PPE, plastic, and other waste materials as specified by the waste management plan, Statement of Work, or other applicable documents.

5.0 RECORDS

5.1 General Requirements

5.1.1 Records generated as a result of this procedure or reference procedure(s) shall be processed and maintained in accordance with requirements in BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*].

5.2 Quality Records

- Field logbooks
- Chain-of-custody forms

5.3 Non-Quality Records

- None

6.0 SOURCE DOCUMENTS

- U.S. Environmental Protection Agency (EPA), November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual,* Section 11. Region 4, Environmental Compliance Branch, Athens, GA.
- Portland State University, Ecosystem Research, Ecological Knowledge, Qualitative Modeling, *Measuring Tree Age,* Retrieved January 11, 2007 from [http://cse.pdx.edu/forest/forest_tree_ages.htm]

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 1

DEFINITIONS

AUGER – A hollow drill, part of the tree increment borer, that when advanced into a tree, forces a core of wood into its interior.

HANDLE – Part of a tree increment borer that fits together with the auger to form a T-shaped hand drill.

SPOON – Part of the tree increment borer used to remove the wood core by sliding between the core and the inner surface of the auger then extracting (also called the extraction tool).

TREE INCREMENT BORER – A tool that includes a handle, an auger, and an extraction rod used to take a sample by boring through a tree and extracting the core.

ACRONYMS

BJC – Bechtel Jacobs Company LLC
DOE – U.S. Department of Energy
EPA – U.S. Environmental Protection Agency
FSP – Field Sampling Plan
PGDP – Paducah Gaseous Diffusion Plant
PPE – personal protective equipment
PRS – Paducah Remediation Services LLC
SAP – Sampling and Analysis Plan

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Environmental Monitoring	**PRS-ENM-1001**	**REV. NO. 0**
SUBJECT MATTER AREA: Environmental Data; OREIS	**PREPARER: Lisa Crabtree**	**Page 1 of 4**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** **Tracey Brindley (Signature on File in DCC)** **2/26/07**	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **TRANSMITTING DATA TO THE PADUCAH OAK RIDGE ENVIRONMENTAL INFORMATION SYSTEM (OREIS)**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 3/05/07**	
USQD/UCD No: USQD-PH-SITE-0031/UCD-PH-SITE-0065	**REQUIRED REVIEW DATE: 3/05/10**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of Paducah Remediation Services documentation. This document replaces BJC-ES-1001, Rev. 0, *Transmitting Environmental Data to the Oak Ridge Environmental Information System (OREIS)*.	All

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3.3 Data Review and Standardization Process 3
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1.0 PURPOSE

This procedure describes the responsibilities and action steps for transmitting data including, but not limited to, analytical results, field measurements, and location data, to Paducah OREIS.

2.0 SCOPE

This procedure shall be used for the transmittal of data to Paducah OREIS by PRS and subcontractor personnel. This procedure describes (1) transmittal of data to Paducah OREIS by data provider, (2) review and standardization of transmitted data by Paducah OREIS staff, (3) review and authorization for release of data via Paducah OREIS by data provider, and (4) public release of data.

This procedure does not cover any data activities that are conducted after the data have been loaded into Paducah OREIS.

3.0 PROCEDURE

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by PRS.

3.1 Data Requirements

- Data supporting regulatory reports are due to OREIS before the date the report is due to the regulators.

- Data must meet uncontrolled, unclassified, clearance requirements (i.e., must be cleared for public release). Evidence of this clearance status must be provided on the *Paducah Data Release to External Agencies* form, ENM-F-0021. This form is an attachment contained in PA-5003, *Quality Assured Data - Paducah* (PRS-ENM-5003, *Quality Assured Data)*.

- Project measurements and analytical data transmittal must be consistent with the OREIS Data Dictionary and the RTL format. These documents can be obtained from the Data Manager.

- Project measurements and analytical data must be transmitted in electronic format as TXT files.

Data Coordinator

3.2 Data Transmittal Process

1. Ensure that project measurements and analytical data are consistent with the latest revision of the OREIS Data Dictionary and the RTL and that the *Paducah Data Release to External Agencies* form, ENM-F-0021 has been completed before

proceeding.

2. **IF** data will be released to the regulators or the public,
   THEN ensure the data have had the appropriate reviews and approvals (as evidenced by a signed *Paducah Data Release to External Agencies* form, ENM-F-0021 and a completed *Data Assessment Review Checklist*, ENM-F-0020).

3. Coordinate the submission of the data to Paducah OREIS with other data management staff, if necessary.

Data Management Staff

3.3 Data Review and Standardization Process

1. Receive and review RTL file.

2. Process the data and work with Data Coordinator as needed to address data inconsistencies.

3. Complete data processing and notify Data Coordinator that data set has been loaded to Paducah OREIS.

4. Review the data assessment package and file a copy of the *Paducah Data Release to External Agencies* form, ENM-F-0021, in the project files.

4.0 RECORDS

The record copy of the data assessment package, which includes all of the original forms referenced in this procedure, is a QA record and shall be maintained according to BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

5.0 SOURCE DOCUMENTS

Federal Facility Agreement for the Paducah Gaseous Diffusion Plant

Attachment A
ACRONYMS

ACRONYMS

OREIS – Oak Ridge Environmental Information System

PRS – Paducah Remediation Services, LLC

QA – Quality Assurance

RTL – Ready-to-load

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Environmental Monitoring	**PRS-ENM-1002**	**REV. NO. 0**
SUBJECT MATTER AREA: Environmental Data; OREIS	**PREPARER: Lisa Crabtree**	**Page 1 of 4**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** **Tracey Brindley (Signature on File in the DCC)** **2/26/07**	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY:**		
TITLE: **SUBMITTING, REVIEWING, AND DISPOSITIONING CHANGES TO THE ENVIRONMENTAL DATABASES (OREIS AND PEMS)**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 3/05/07**	
USQD/UCD No: USQD-PH-SITE-0031/UCD-PH-SITE-0065	**REQUIRED REVIEW DATE: 3/05/10**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of Paducah Remediation Services documentation. This document replaces BJC-ES-1002, Rev.1, *Submitting, Reviewing, and Dispositioning Changes to the Environmental Information Management (EIM) databases (OREIS, PEMS and TRACKER)*	All

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3.3 Paducah OREIS Change Request 3
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1.0 PURPOSE

This procedure describes the required actions for submitting, reviewing, and dispositioning proposed changes to the PRS Environmental databases. These databases are the Paducah OREIS and PEMS.

2.0 SCOPE

This procedure establishes both the configuration control and configuration management processes of OREIS and PEMS that involve submission, review, and disposition of a change request. It does not cover the subsequent development, QA, testing, and implementation. This procedure applies to (1) OREIS and PEMS data management staff, users, and data providers who request changes; and (2) the individuals who are involved in the review and disposition processes as outlined in this procedure. Change requests to the databases may involve the following: (1) the commercial hardware and software; (2) the user interface and customized software (program changes); (3) the data model; (4) the data values; (5) the reference tables; and (6) documentation.

3.0 PROCEDURE

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by PRS.

3.1 Requester Information

Requester

3.1.1 Submit an OREIS or PEMS change request to the Data Manager. Requests may be submitted via e-mail, telephone, or fax.

3.1.2 Include the description of the change, the affected database (OREIS or PEMS), and if the request is a “Rush.”

3.2 PEMS Change Request

Data Manager

3.2.1 Review PEMS Change request with other data management staff, as needed.

3.2.2 Determine if change requires updates to the Paducah PEMS configuration structure and/or user’s guide.

3.2.3 **IF** PEMS change request is rejected, **THEN** notify the requester of the change request rejection.

3.2.4 **IF** PEMS change request is approved, **THEN** implement change(s) and notify PEMS users of implemented change.

3.3 Paducah OREIS Change Request

Data Manager

3.3.1 Review Paducah OREIS Change request with other data management staff, as needed.

3.3.2 Determine if change requires updates to the Paducah OREIS Data Dictionary and/or the Paducah OREIS User Guide.

3.3.3 **IF** Paducah OREIS change request is rejected, **THEN** notify the requester of the change request rejection.

3.3.4 **IF** Paducah OREIS change request is approved, **THEN** forward to Data Management staff for processing.

Data Management Staff

3.3.5 Process approved Paducah OREIS change request utilizing the X_ACTION queries and tables in the OREIS database, as needed.

NOTE 1: The X_ACTION tables are used to store an original copy of the records before they are updated. The X_ACTION tables contain a detailed description of the changes along with the date the changes were implemented. This allows the Data Management staff to access the original records, if needed.

3.3.6 Notify all Paducah OREIS users of implemented change request.

NOTE 2 : Any changes that affect the Paducah OREIS web application will be incorporated on the next scheduled update**.**

4.0 RECORDS

Records generated by this procedure shall be maintained according with BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

5.0 SOURCE DOCUMENTS

none

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 1

DEFINITIONS

Commercial hardware/software – Products purchased from commercial vendors and integrated into OREIS or PEMS that includes, but is not limited to, NT servers, UNIX server, operating systems, a relational database management system, or a geographic information system.

Configuration control – A process that involves establishing the baseline computer system and controlling system changes thereafter to be consistent with approved prioritization, quality, and budget parameters; the systematic evaluation, coordination, implementation, and documentation of all changes to a system after its completion

Configuration management – Conducting configuration control of the Environmental databases as prescribed in this procedure, the OREIS Charter, and the OREIS and PEMS users guides.

Data Model – Representations of the data relevant to OREIS and PEMS and their interrelationships. The data model is a logical representation of the database structure and is used to develop the physical database structure.

Data Manager – The staff member responsible for overseeing the implementation of changes to data and database structures approved by this procedure.

Requester – Person submitting a change request.

ACRONYMS

DOE – U.S. Department of Energy

Paducah OREIS – Paducah Oak Ridge Environmental Information System

PEMS – Project Environmental Measurements System

PRS – Paducah Remediation Services, LLC

QA – Quality Assurance

USQD – Unreviewed Safety Question Determination

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date **April 24, 2006**

PROCEDURE TO BE TRANSITIONED:

Functional- Environmental Monitoring

Procedure Type
(Company, Functional, Project, Subcontractor)

ES-1510 ~~BJC-ES-01,~~ **Subcontracting Analytical Work and Sample Material Tracking**

(Procedure Number and Title) **(ES-1510)**

6-21-00
(Procedure Date)

2
(Rev. Number)

Reason for Changes: **Transition from BJC to PRS Paducah Remediation Contract**

SECTION	DESCRIPTION OF CHANGE
All	Replace BJC positions/titles/organizations with PRS positions/titles/organizations as indicated on attached crosswalk/matrix
All	Change text references to BJC or Bechtel Jacobs Company to PRS or Paducah Remediation Services with the exception of procedure titles and procedure numbers.
All	Change SMO Manager to Environmental Monitoring/Sample Mgr
All	Change SMO APM to Sample Handler/Lab Coordinator
All	Change SMO Technical Procurement Specialist to Laboratory Coordinator
All	SMO Technical Invoicing Specialist to Laboratory Coordinator
All	Technical Procurement Specialists to Procurement Representative
All	SMO Transportation Management to Laboratory Coordinator
Attachment A	Change BJC acronym to PRS
Title Page	Page block should read 1 of 15 not 1 of 46

Initiator/Originator: **Tom Barron** Tom Barron **4-19-06**
(Print Name / Sign Name) Date

REVIEWED BY:
Carleton Edmunds **4-19-06**
Discipline/Task Manager Date

APPROVED BY: **Carleton Edmunds** **4-19-06**
Date
Project Manager

JJ 4-19-06 **BJC to PRS Position/Title/Organization Matrix**
for {~~BJC-ES-01~~ ES-1S10 Subcontracting Analytical Work and Sample Material Tracking}

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
SMO Manager	→	Environmental Monitoring/Sample Mgr
SMO APM	→	Sample Handler or Lab Coordinator
SMO Technical Specialist	→	Laboratory Coordinator
Technical Procurement Specialist	→	Procurement Representative
SMO Transporation Management	→	Laboratory Coordinator
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BECHTEL JACOBS
Bechtel Jacobs Company LLC

BJC-ES-01, Environmental Services Procedures	ES-B-0410	REV. NO. 2
TITLE: SUBCONTRACTING ANALYTICAL WORK AND SAMPLE MATERIAL TRACKING	EFFECTIVE DATE: 6/21/00	Page 1 of 46
	PREPARER/POC: Rick Brooksbank	
	APPROVED BY/DATE: J. W. Goskowicz 3/30/00 [Approval Signature on File]	

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REVISION LOG		
Revision Number	Description of Changes	Pages Affected
1	Performance Indicator Factor (PIF) Revision. Replaced Revision 0 with Revision 1.	Page 3
2	Non-intent change. Revised to incorporate ES-B-0407 requirements. Replaces ES-B-0407, *Sample Management Office Material Tracking Process.*	All

PURPOSE

This procedure describes the methodology used by the Sample Management Office (SMO) in processing Analytical Statements of Work (SOWs), selecting appropriate analytical laboratory support, and implementation of the procurement processes. In addition, this procedure describes the process for tracking samples subcontracted through the SMO.

SCOPE

This procedure applies to all Bechtel Jacobs Company LLC (BJC) organizations requiring services from the SMO, SMO personnel, project and program personnel, and commercial laboratory personnel responsible for managing samples from collection, through shipping (if applicable), to final disposition.

TRAINING

None

OTHER DOCUMENTS NEEDED

- BJC Organizational Chart
- K/DSRD-3022/D, *Oak Ridge Sample Management Office TRACKER User's Manual for Customers*
- K/DSRD-3020/D, *Oak Ridge Sample Management Office (SMO) TRACKER User's Manual for SMO Users*

RESPONSIBILITIES

SMO Manager

- Ensure SMO Analytical Project Managers (APM) are trained to this procedure.
- Ensure SMO APMs are up-to-date with TRACKER, Contract Compliance Verification, and process is completed real time.
- Provide adequate systems resources such as personal computers, fax machines, and TRACKER.
- Ensure SMO APMs are provided technical training in respective areas.
- Ensure coordination of process flow.
- Ensure adequate administrative support.
- Ensure adequate laboratory resources are available to provide capacity and capability for project needs.

SMO APM

- Work with projects.
- Enter information on TRACKER.
- Request spot quotations.

	• Ensure selection of laboratory.
	• Review invoices.
SMO Technical Procurement Specialist	• Assist the SMO APM with TRACKER data entry when necessary.
	• Prepare project files.
SMO Technical Invoicing Specialist	• Perform purchase modifications.
	• Distribute invoices.
	• Submit the notice of completion.
	• Initiate de-commit actions and completion
	• Prepare completed project files for transfer to records holding.

WHAT TO DO

A. Subcontracting Analytical Work

SMO Manager	**1.** Assign a SMO APM to individual projects to assist with planning, phasing, and execution of projects.
SMO APM	**2.** Identify the project's analytical requirements.
SMO Technical Procurement Specialist	**3.** Begin preparing a project file and ensure required documentation is added throughout the project.
SMO APM	**4.** Assist projects by providing input to data quality objectives, quality assurance plans, project plans, and sampling and analysis plans.
	5. Develop or assist in developing an analytical SOW on TRACKER for the execution of the project. Ensure the third page of TRACKER is signed by the project leader.
	6. Provide analytical cost estimates and data to project.
	7. Review and split project SOW into the following sub-parts: environmental chemistry, radiochemistry, physical measurements, dioxins/furans, as appropriate.

8. Submit a letter, SOW with attachments, and a spot quotation form to the SMO approved government owned-contractor operated (GO-CO) laboratories and commercial laboratories participating in the BJC Analytical Support Agreement to request spot quotations. Alternatively, spot quotations may be requested from specialty laboratories based on specific requirements (Attachment B). In addition, these laboratories can enter their pricing directly into TRACKER.

9. After receiving spot quotations from the laboratories, add the pricing information to the "Laboratory Selection" module on TRACKER.

10. Select a laboratory (or laboratories), giving first consideration to the recommended "best value added" on TRACKER.

"Best value added" is an assessment of total project price which is total price is laboratory estimated cost plus the estimated shipping cost to that laboratory per performance. It is calculated as below:

Performance Indicator Factor (PIF) = (TAT x 60) + (HT x 20) + (CV x 10) + (PE x 10) where TAT is turnaround time performance for past ninety days, HT is holding time performance for past ninety days, CV is contractual verification for past ninety days and PE is both single blind and double blind performance evaluation performance during past 360 days. "Best Value Added" is lowest ratio of total price divided by PIF.

Project or SMO APM

11. IF sufficient justification exists to request a waiver on the selection process, **THEN**
obtain concurrence by the SMO Manager and the Manager of Sample and Data Management.

SMO APM

12. IF a waiver is received, **THEN**
enter a narrative on the comment section of "Laboratory Selection" Module on TRACKER.

SMO

13. Create an Intent to Offer letter.

Manager of Sample and Data Management, or designee

14. Sign the Intent of Offer letter.

SMO

15. Submit the Intent to Offer letter to the project's selected laboratory.

OR

Technical Procurement Specialist

16. Fax the Intent to Offer letter to the selected laboratory for projects <$25,000.

17. Provide backup required documentation [SOW with attachments, comparison of offers (detail and summary), spot quote offers] to Procurement.

18. Once acceptance has been received from the laboratory, enter the requisition onto BJC Automated Procurement System (APS).

The hierarchy of approvals is as follows: SMO Manager, Safety, and Sample & Data Management Manager, Projects Controls, and Project Manager. As an alternative, a purchase memorandum signed by the project controls engineer and project manager (with appropriate Policy 230 signature authority) can be used for approvals listed for those positions in APS. However, the information must still be entered into APS.

19. Enter requisition number, period of performance, and purchase order amount in the "Laboratory Selection" Module on TRACKER.

SMO APM

20. Notify the project of selected laboratory via e-mail from TRACKER.

21. Hold a readiness review with the laboratory and project, usually by conference call. Document the readiness review on TRACKER.

22. During ongoing project activities, ensure the following information is entered on TRACKER:

- sampling data
- shipping data
- results
- approved invoices.

SMO Technical Invoicing Specialist

23. IF requested, **THEN**
perform purchase order modification requests for increase/decrease in funding or performance period.

SMO Manager

24. Receive invoices from the laboratory (or laboratories) and submit to the SMO Technical Invoicing Specialist.

SMO Technical Invoicing Specialist

25. Distribute invoices to appropriate SMO APMs for approval.

SMO APM

26. Recommend or reject the invoice. Submit approved invoices to the Technical Invoicing Specialist.

27. IF the invoice is rejected, **THEN**
resubmit to the laboratories for correction.

SMO Technical Invoicing Specialist

28. Enter the date of approval on TRACKER.

29. Submit the approved invoices to the Laboratory Subcontractor Technical Representative who will review, sign and forward the invoice to Accounts Payable.

SMO APM

30. After project is completed, de-commit excess funds when necessary.

SMO Technical Invoicing Specialist

31. After the project is completed, submit the notice of completion to respective laboratories for concurrence and acknowledgement of completion.

32. Upon receipt of laboratory agreement and concurrence, initiate de-commit actions and completion on APS and code TRACKER as complete and file project folder in completed file.

33. Prepare completed project files and transfer to records holding as required.

B. Sample Material Tracking

SMO Customer/Project

1. Submit SOWs to the SMO and ensure they contain the information needed for evaluation of shipping concerns.

2. Notify SMO Transportation Coordinator of sample collection schedule for submission of samples to commercial-sector laboratories.

3. Supply SMO with chain-of-custody (COC) forms and any other supporting documentation for samples being transported or shipped.

4. Ensure that the samples collected and the analyses requested adhere to the SOW as issued to the laboratory.

5. **IF** the number of samples changes or the requested analysis changes, **THEN**
prepare an amended SOW and notify the SMO APM prior to the shipment of any samples.

6. Notify the SMO Transportation Coordinator of impending sample shipment.

7. Supply the SMO Transportation Coordinator with a copy of the COC and any associated documentation (i.e., radiological activity report).

SMO APM

8. Complete the SOWs, which reflects the number of samples to be collected, the sampling time frame, the requested analyses, any historical information, and the desired disposition of the samples.

9. Identify which projects will submit samples to commercial facilities and which projects will submit samples to GO-CO internal and external laboratories.

10. Interface with laboratories to schedule sample shipment and residual returns.

11. Maintain copies of shipping records associated with all shipments of samples processed through the SMO.

12. Ensure that all data pertinent to sample shipment and disposition are updated in the TRACKER system.

13. Supply the analytical laboratory a copy of the COC, precautions, and additional instructions as required.

14. Supply the laboratory a copy of the radiological report is the samples contain radiological constituents.

15. Request the commercial laboratory designated to receive radiological samples to review and compare their radiological license values against the radiological activity report and fax to the SMO Transportation Coordinator a written permission to ship samples.

NOTE: This is not applicable to GO-CO laboratories.

Laboratory

16. Supply the SMO with a copy of the COC shipped with the samples, sample receipt form, and a copy of the laboratory sample log-in.

17. Supply the SMO with a copy of waste management documentation including the batch number for a sample group, waste manifest, waste shipping papers, disposal company name and address, and certification of disposal.

18. Review radiological license, if requested, and fax written permission to ship specific sample group.

19. Complete sample receipt form when sample group is received.

20. Sign the COC received with the sample group.

21. Log samples into the laboratory database.

22. Fax a copy of the COC, sample receipt form, sample log-in to the SMO Transportation Coordinator.

23. Provide the SMO Transportation Coordinator with a copy of the sample disposition documentation for samples designated to be disposed by the commercial laboratory (sample group waste stream batch numbers, waste manifest, disposal company name and address, and certification of disposal).

24. Provide the SMO Transportation Coordinator with written verification of samples consumed in analysis.

25. Return sample residues to the SMO Customer when requested.

SMO Transportation Management

26. Review the SOW and identify concerns for shipping, disposition, and tracking.

27. Update the shipping files with information provided by the laboratory.

28. Notify the laboratory, before end of archival, with logistics for sample residues to be returned to the SMO Customer.

29. Update disposition files and update TRACKER with sample disposition information.

RECORDS

All sample tracking information is maintained on TRACKER; no record copies are generated as a result of this procedure. Documentation of sample disposition is held by the commercial laboratory - copies are included in the shipping file for the specific SOW.

Shipping Department

- Maintain sample shipping order records.

SOURCE DOCUMENTS

None

Attachment A
ACRONYMS/DEFINITIONS
Page 1 of 1

APS – Automated Procurement System

BJC – Bechtel Jacobs Company LLC

DOE – Department of Energy

COC – chain-of-custody

GO-CO – Government owned-contractor operated. This applies to prime DOE contractors.

Sample Management Office (SMO) – A group within Sample and Data Management tasked with providing direct support for Analytical Services to BJC.

TRACKER – An automated system (database) which defines deliverables, cost, quotes, performance, and project accounting.

Customer – An environmental project or program requesting services from the SMO.

Waste Manifest – A document initiated for transporting regulated wastes which describes and inventories the waste.

Statement of Work (SOW) – A contractual document specifying the analytical requirements for a given project. Customers generate SOWs electronically on TRACKER.

Shipping Papers – A shipping order, bill of lading, manifest, or other shipping document serving a similar purpose and containing the information required by the Department of Transportation for transportation of a hazardous material in commerce.

Attachment B
EXAMPLE SPOT QUOTE LETTER
Page 1 of 1

Bechtel Jacobs Company LLC
Melanie Underwood
Sample Management Office
Post Office Box 4699
Oak Ridge, Tennessee 37831 - 7169
Facsimile: 423-574-9433

BECHTEL JACOBS
Bechtel Jacobs Company LLC

April 19, 2006

Participating Oak Ridge Sample Management Office Environmental Chemistry Laboratories

SPOT QUOTATION for Roof Removal Project SOW # BKET0857
The Sample Management Office is seeking spot quotations from approved Pricing Agreement laboratories for the above referenced project. The spot quotations are specific only for the above project and your response and pricing is not constrained by the previous prices submitted under the Pricing Agreement. All terms and conditions shall apply except as modified in the statement of work. The award will be made on an "all or none" basis.

This project may have requirements not covered in the Pricing Agreement. This invitation is being sent to all environmental chemistry approved Pricing Agreement laboratories. Pricing for this spot quotation **will not** be added to the Pricing Agreement, as this is a project-specific spot quotation. The pricing submitted should consider the entire project requirements as to samples and shipping containers, methods, required quantitation limits, quality control, turnaround times, and data deliverables. Under the terms and conditions of the pricing agreement a **penalty of 2% per day** will be assessed on deliverables (i.e., data packages plus EDD, if required) received beyond the turn around time.

SOW- BKET0857 is enclosed for your evaluation along with a consolidation of SOW- Attachments. Your quotation should include a price per sample per the enclosure. The number of samples indicated in the enclosure is an estimate and may not represent the actual number of samples submitted. However, the award will be based on the extension of the estimated number of samples multiplied by the quoted price. As always, the award will be offered to the laboratory that meets the technical requirements of the project and provides the "best value added" to the project, where "best value added" is a composite score based on pricing and performance. The laboratory must confirm or reject the offer without changing any pricing.

All responses, which are to be considered for this project, must be received in this office by **12:00 NOON EST, Thursday, August 6, 1998.** As always, I am available to answer any question you may have on this project (423-241-1368). Thank you for your continued cooperation and responsiveness.

Sincerely,

Sample Management Office

Enclosures
By Fax
cc: File-SMO-RC

Attachment C
EXAMPLE INTENT TO AWARD LETTER
Page 1 of 1

Bechtel Jacobs Company LLC
James A. Ealy
Sample Management Office
Post Office Box 4699 Oak Ridge, Tennessee 37831 - 7169
Telephone: 423-576-2724 Facsimile: 423-574-9433

Comment [Rd1]: After opening the form the cursor will be located at an insertion point directly under □Lockheed Martin Energy Systems. At the insertion point, type in your building, mailstop and room number. After inputting the information, use the tab key to advance to the next point of insertion and input the post office box, tab again and input the four digit zip code; tab a final time and input the area code and telephone number. AFTER TYPING IN THE TELEPHONE NUMBER, TAB ONCE AND CLICK ON THE NEXT RED BUTTON AND READ INSTRUCTIONS BEFORE PROCEEDING!!!!

Comment [Rd2]: After reading the information in this help box, you will input the area code and fax number (it is O.K. to type on top of the red icon). After typing in the facsimile number, **DO NOT USE THE TAB KEY!!**. Use your mouse to click on the next red comment box located in the left margin. Read and follow the instructions inside the box to complete the body of your letter. **Using Tab key may insert additional rows.**

October 22, 1998

Dear Mr. :

SOW BXPT00915

The Sample Management Office (SMO), on the behalf of Bechtel Jacobs Company LLC, is pleased to inform you that it is the intent of the Company to award to you the subject statement of work based on your spot quotation for the project. Project work shall not begin until a subcontract/purchase order has been issued by the Company. Please confirm the project price of $_____on the estimated number of samples to be submitted and your review of the complete statement of work. Please note that your laboratory was provided a copy of the statement of work (SOW) prior to receiving your quotation. The SMO recognizes that the actual number of samples submitted may vary from the estimate and will approve remittance based on your quoted unit price and the actual number of samples submitted. As previously stated, the terms and conditions of the Pricing Agreement remain in effect except for the pricing received as a project specific quotation.

The SMO would like to receive your acceptance confirming the pricing or declination of this SOW within 24 hours of receipt of this fax. As always, I am available to answer any technical questions related to this project.

The SMO appreciates the continued cooperation and support of your laboratory. Please contact _____ at _____, if you need additional information.

Sincerely,

James A. Ealy
Manager
Sample Management Office

JAE:sar
By Fax (1 page)
cc: File-SMO-RC

Attachment D
EXAMPLE PROJECT OR PROGRAM LETTER
Page 1 of 2

FROM: **Oak Ridge Sample Management Office**

TO: **Project/Program Manager**

RE: **Coordination of Sample Transportation for Statement of Work (SOW)**

Thank you for giving the Oak Ridge Sample Management Office (SMO) an opportunity to provide your analytical needs.

The Department of Energy (DOE) has tasked the Oak Ridge Sample Management Office Transportation Coordinator with providing oversight for sample shipping and transport activities, sample tracking, performance evaluation sample (PE) coordination, and tracking sample disposition status.

The SMO requires that all samples shipped to external laboratories be coordinated through the SMO Transportation Coordinator. Procedure *ES-B-0410* identifies the steps for tracking samples brokered by SMO.

To comply with the DOE directives and provide the SMO with the necessary information for the shipping and tracking processes, the SMO Customers must supply specific information and documents to the Transportation Coordinator for sample shipping and tracking activities.

- Provide information to the Transportation Coordinator for return of shipping package (e.g. coolers, drums, and cartons).
- Identify a mode of sample disposition (return unused sample, lab disposes unused sample, or sample will be consumed in analysis). This information may also be included in the SOW.
- Provide any special instructions to be transmitted by the Transportation Coordinator to the laboratory. The Transportation Coordinator will interface with the laboratory for the SMO Customer for sample shipping and tracking concerns.

For samples shipped through site shipping departments the SMO Customer:

- Request the Transportation Coordinator issue a shipping order for samples going to an off-site laboratory.
- Supply the Transportation Coordinator with a copy of the Chain of Custody (COC) for the samples to be shipped, a copy of the Request for Analysis (RFA) when not included on the COC, and a radiological activity report when samples include radiological constituents. The COC and RFA may be generated electronically in TRACKER and available to the SMO.

Attachment D
Page 2 of 2

For samples collected and shipped from the field the SMO Customer:

- Provide the Transportation Coordinator with a tentative shipping schedule and notify the Transportation Coordinator prior to shipment of samples and transmit, as soon as feasible, a COC, RFA, and radiological activity report if applicable. The minimum document required is a COC. The COC and RFA may be generated in TRACKER.

The Analytical Support Division has an shipping group trained and available to help SMO Customers with Classification, packaging, labeling, marking and preparing shipping papers should the need arise for these services.

The goal of SMO is to provide assistance to our customers for their analytical needs in a timely and cost effective manner. It is the intent of the SMO to provide as many services as needed to ensure the success of DOE-ORO funded projects and programs. If you need further help, please contact my office (423) 576-7381, or page me (423) 873-4387.

Thank You,

B.J. Scott
OR-SMO Transportation Coordinator
K-1001, MS-7169

Attachment E
EXAMPLE COMMERCIAL LABORATORY LETTER
Page 1 of 1

OAK RIDGE SAMPLE MANAGEMENT OFFICE
Sample Transportation Coordination
P.O. Box 2003, K-1001, MS-7169
Oak Ridge, TN 37831
Phone: (423) 576-7381
Fax: (423) 574-9433

TO: **Laboratory Name**

FROM: **Oak Ridge Sample Management Office**

RE: **Sample Shipments and Sample Tracking**

This communication pertains to SOW-XXX. The award of this work is governed by the Terms and Conditions and the requirements of the SOW. This letter further clarifies elements needed by the OR-SMO for sample accountability and tracking.

Laboratory

Return by fax, same day, a copy of the COC for samples shipped, sample log-in ID numbers assigned by the laboratory, and a copy of the sample receipt form as samples are received.

For samples containing radiological or suspect of containing radiological constituents the laboratory will be requested to review an activity report, compare the activity report to their rad license, and after reviewing the activity report, fax the SMO permission to ship for samples of concern.

Provide the Transportation Coordinator with documentation for tracking unused sample to disposal (waste batch numbers/sample group, manifest numbers/sample group, and certification of disposal/sample group).

Review and comment any special instructions or precautions attached.

The OR-SMO will contact the laboratory prior to each shipment and notify of pending arrivals. In the notification the sample ID, sample type, sample matrix, and number of containers will be provided. When possible a copy of the COC will also be included in the notification. For shipments that contain radiological contaminants, an activity report will also accompany the notification for review by the laboratory.

Attachment F
SAMPLE RECEIPT FORM
Page 1 of 1

OAK RIDGE SAMPLE MANAGEMENT OFFICE

SAMPLE RECEIPT NOTIFICATION

Receiving Laboratory: _____

Name of individual receiving sample shipment: _____

SOW Number: _____Today's Date: _____Time: _____(military)

Shipping number assigned by the carrier:_____

Condition of shipping container:Good__Fair___Poor__Damaged
If damaged please describe:_____

_____

Custody seals intact on shipping container: _____Yes _____No

Received within acceptable holding times: _____Yes _____No

Samples preserved: _____Yes _____No (Chemical Chilled None)

COC accompanied samples:___Yes _____No

Discrepancies between COC and Samples:_____Yes___No
If yes please explain_____

_____

Other documents accompanying shipment:_____RFA__Activity Report _____Precautions___Special Instructions_____Other _____

Temperature of samples as received:_____°C.

Sample containers intact:_____Yes___No

When samples are received complete and fax this form, same day, to B.J. Scott, fax number (423) 574-9433. Questions, call Mr. Scott at (423) 576-7381 or page at (423) 873-4387.

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PGDP Procedure BJC-ES-2000

Field Screening Using an INNOV-X Systems Hand-Held X-Ray Fluorescence Spectrometer

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Environmental Monitoring	**PRS-ENM-2300**	**REV. NO. 0**
SUBJECT MATTER AREA: Engineering	**PREPARER: K. Holland**	**Page 1 of 18**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** **Tracey Brindley (Signature of File in DCC)** **3/12/07**	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **COLLECTION OF SOIL SAMPLES**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 3/19/07**	
USQD/UCD No: USQD-PH-SITE-0039 & UCD-PH-SITE-0078	**REQUIRED REVIEW DATE: 3/19/10**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of PRS. This document replaces BJC-ES-2300, *Collection of Soil Samples*, Rev. 0; GEO-TEC-015, *Subsurface Soil Sampling*, Rev. 0; GEO-TEC-020, *Surface Soil Sampling*, Rev. 2; W-157-PWOS, *Surface Soil Sampling*, Rev. 1; and W-158-PWOS, *Subsurface Soil Sampling*, Rev. 1.	All

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1.0 PURPOSE

This procedure describes the standard methods and equipment needed to collect environmental or geotechnical soil samples for description, physical and/or chemical analyses. The sampling methods as follows:

- Trier, spade, spoon, trowel or scoop;
- Stainless steel hand-operated auger;
- Thin-walled sampler; and
- Split-barrel sampler.

The task-specific WP will specify the type of soil sampler(s) that will be used to collect disturbed or undisturbed soil samples at the surface, from boreholes, or test pit/trench/excavation/trench sidewalls or bottoms. The manual or powered equipment and methods used to reach or access and sample soil at the surface and in the subsurface are dependent upon project objectives; soil conditions (i.e., degree of consolidation and moisture content; type; volume and depth of sample required; and the characteristics of the soil to be sampled) and should be specified in the WP. The method used can be either manual (e.g., drive hammer, hand auger, post-hole digger, and slide hammer) or powered (e.g., drill rig, direct push, pneumatic hammer, manually controlled mechanical or hydraulic earth drills, conventional excavation equipment).

2.0 SCOPE

This procedure shall be used for project characterization and cleanup verification activities performed by PRS.

3.0 PROCEDURE

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by PRS.

3.1 Quality Assurance/Quality Control

1. QC Samples

   The number and types of QC samples (e.g., duplicate, equipment rinsate, trip blank, etc.) shall be collected as specified in the task-specific WP and prepared in accordance with BJC-ES-2704, *Trip, Equipment and Field Blank Preparation* (PRS-ENM-2704, *Trip, Equipment and Field Blank Preparation*). Rinsate blanks are not required when disposable sampling equipment is used.

2. Control and Documentation of Deviations

   Any departures from the requirements specified in the approved,

task-specific WP or this procedure shall be authorized through the PRS Project Manager (or designee). Prior to deviating from those requirements, authorization and WP revision shall be in accordance with BJC-FS-1001, *Work Control Process* (PRS-WCE-0018, *Paducah Environmental Remediation Project Work Management Program*). Any deviations and one-time difficulties encountered in the field concerning sample collection or related activities shall be documented in the field logbook by the Sample Handler.

3.2 Field Preparations

1. PEMS is the data management system that supports sampling and measurement collection activities, the automatic transfer of laboratory SOW and COC information, and the receipt of analytical results in digital format.

   PEMS is the preferred approach for generating sample container labels and COC forms. The sampling team will pre-populate the PEMS database in accordance with the project-specific requirements.

2. The sample handler shall prepare field logbook(s) in accordance with BJC-ES-2700, *Field Logbooks* (PRS-ENM-2700, *Logbooks and Data Forms*).

3. The sampling team will assemble the necessary equipment, tools and supplies, and ensure that all required notifications, permits [e.g., RWP (if required), excavation and penetration permit, burn permit, hot-work permit] are active and in place, and that all pre-mobilization readiness activities are complete prior to field mobilization.

4. Sample technicians, sampling tools, and equipment will be obtained decontaminated and ready for use [see BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* (PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*)] and shall be protected from sources of contamination during transport to the sampling location and between each individual sampling location during field sampling activities (e.g., wrapped in aluminum foil, sealed in plastic or other storage containers, placed on plastic sheeting in staging area).

5. Disposable sampling implements and supplies shall be new and unused and will not be decontaminated for reuse, but will be disposed of in accordance with WP and PRS waste handling procedures

6. Obtain new and certified clean sample containers from a commercial supplier or from the supporting analytical laboratory.

7. Enter the sample container lot number in the field logbook.

3.3 Equipment, Tools, and Supplies

The sample handler is accountable for preparing and gathering all needed logbooks, field instruments, samplers, equipment, tools and supplies to safely and successfully complete the soil sampling event. The items listed may be used as a guide, but may not be a complete list. Refer to the task-specific AHA(s) and WP, and the RWP (if one is required) to determine what instruments, supplies, materials, samplers and equipment are needed to safely execute the activities comprising the soil sampling event. Additional method specific requirements are included in the procedure methods of Section 3.4 through 3.6.

1. Field logbook(s)
2. PPE and other safety equipment specified by the AHA and RWP
3. Task-specific ES&H supplies needed to establish and control the work area
4. COC forms generated from PEMS database
5. Sample labels generated from PEMS database including extra labels
6. Custody seals
7. New sample containers appropriate for the samples to be collected with COA, or certificates of cleanliness
8. Survey pin flags, stakes or paint
9. Traffic control vests, cones and signs
10. Duct tape
11. Ruler and tape measure
12. Utility knife
13. Plastic bags and plastic wrap
14. Aluminum foil
15. Cooler chest(s) with ice or blue ice.
16. Decontamination supplies and equipment
17. Stainless steel or Teflon®-coated spade
18. Stainless steel, glass, Teflon®or disposable bowls
19. Stainless steel, Teflon®-coated or Teflon®trowels, scoops, scoopulas,

spoons, and/or spatulas

20. Teflon®sheet/film

21. Silicone tape or other U.S. EPA-approved sealing tape

22. Sample tubes and end caps

23. Indelible marking pens

24. Analytical balance

25. The following sampler types, with needed ancillary equipment and supplies as required by the sampling method specified in the WP:

- Spade, Spoon, or Scoop;
- Stainless steel hand-operated auger;
- Split-barrel sampler (with or without inserts);
- Thin-walled sampler and tubes;
- EnCore®or equivalent sampler and containers; and
- Trier

3.4 General Requirements

The sampling team members as assigned by the Project Manager (or designee) shall complete the following steps prior to initiating the soil sampling event.

1. Review the WP for the sampling method(s) and equipment to be used and reference to any photos, maps or figures that indicate or show proposed soil sampling locations, method(s) to be used, type(s), and number of soil samples to be collected (e.g., discrete or composite).

NOTE 1: Refer to the WP to determine whether the area where soil samples are to be collected is within a building, under slab or paved over. The slab or pavement will need to be penetrated prior to soil sample collection.

NOTE 2: Utility clearance will be required for all locations where excavation or penetration will be accomplished to a depth of greater than 1 foot below ground surface. Utility clearance activities will be completed in adherence to PRS procedures prior to excavating, driving, or drilling with the device to access and collect soil samples.

The area within a minimum 5-foot radius of a proposed

penetration (sampling location) will also be cleared for utilities. If a utility is identified within 3 feet of the proposed sampling location, the location will be moved and the clearance procedure will be repeated at the new location. If it is necessary for a penetration to be located in an area of known or suspected overhead and/or subsurface obstructions, special safety precautions will be implemented as required and all ES&H guidance and procedures that control penetrations shall be followed to prevent accidents.

If refusal is encountered [i.e., the manual or power driven/drilled sampling equipment can no longer be advanced (50 blows with a 140-pound hammer to advance sampler 6 inches)] before the required sample depth is reached, the location will be moved within the circumference of the cleared circle and reattempted. If refusal is encountered after 3-attempts, the location will be abandoned and will not be sampled.

The area where the test pit/trench/excavation will be dug shall be cleared for utilities prior to beginning soil sampling. The test pit/trench location will be relocated (if possible) and cleared for utilities if any overhead or subsurface obstructions are located or suspected. Special safety precautions will be implemented as required and all ES&H guidance and procedures that control digging or excavating in areas where overhead or subsurface obstructions are known or suspected shall be followed to prevent accidents.

Deviations encountered in the field concerning sample location, sample collection, or related activities shall be documented in the field logbook by the Sample Handler.

NOTE 3: Soil samples collected for VOC analysis always will be collected first, regardless of the sampler used, to minimize loss of volatiles due to disturbance and offgassing. The preferred collection method, if the VOC soil sample isn't collected, labeled, and shipped in the sampler (i.e., thin-walled tube or insert), is to use an EnCore® or equivalent sampler.

Use the EnCore® or equivalent sampler and sample container to collect the VOC sample(s) following the current EnCore® or equivalent sample collection and sampling procedure. Standard practice is to collect three EnCore® or equivalent samples to represent one sampling point. Deviation shall be recorded in the field logbook and on the COC in the remarks column.

If coarse-grained soil precludes the use of the EnCore® or equivalent sampler for VOC soil sample collection, then use a clean spoon or spatula and directly transfer the sample into a

sample container. Minimize the headspace by completely filling the container with soil. Note the deviation in the field logbook and on the COC in the remarks column.

2. Initiate the sampling event after completing all required notifications and entering required information in the field logbook prior to mobilizing to the field.

3. Record the area that will be sampled in the field logbook and mobilize to that area.

4. Prepare a decontamination area (if required).

5. Pre-locate proposed sampling point(s) using photos and maps (figures) from the WP for guidance prior to sample collection.

NOTE 4: In most cases, proposed environmental soil sampling locations are chosen prior to the actual sampling event and approximately located using a GPS device or other suitable method, or civil surveyed and temporally marked (e.g., a labeled survey pin flag, survey stake or survey paint). Use the pre-marked location(s) to guide the sampling effort.

6. The RCT and/or ES&H Technician will screen all sampling location(s) prior to sampling and inform the sampling team that it is safe to begin soil sampling.

7. Begin work by preparing the area around the proposed soil sampling location by laying down plastic sheeting or an equivalent material as groundcover to prevent contact with potentially transferable contaminants, spread of IDW, and possible cross contamination.

NOTE 5: Protective groundcover may also be required when powered equipment (e.g., drill rig, direct push rig, pneumatic hammer, manually controlled mechanical or hydraulic earth drills) is the method used to penetrate and reach the required sampling depth and collect the soil samples. Consult the WP for direction.

8. Cut a hole in the plastic sheeting of sufficient size to access the sampling point.

9. Use a stainless steel or Teflon®-coated spade to remove surface vegetation and debris from the sampling location if necessary.

10. Perform the sampling using the method and sampler type specified in the WP, while controlling the spread of IDW.

3.5 Soil Sampling Methods

3.5.1 Soil Sampling with a Trier, Spoon, Trowel, or Scoop

1. For soil sample collection at grade, along test pit/trench/excavation sidewalls or bottoms, use a Stainless steel, Teflon®-coated or Teflon® spoon or scoop to remove the layer of soil that may have been disturbed and in contact with the spade, heavy equipment bucket, or grader blade (reference WP).

2. Stage soil IDW on plastic sheeting when sampling at grade.

NOTE 1: A trowel may be used to cut a block of soil (sample) if the WP requires description and sampling of an undisturbed soil profile. The undisturbed sample will be described in accordance with BJC-ES-2303, *Borehole Logging* (PRS-ENM-2303, *Borehole Logging*) prior to preservation for laboratory testing or disruption and soil sample processing for analysis.

3. Use a decontaminated stainless steel trier [go to Step (4)], or use a Teflon®-coated or stainless steel trowel, scoop, scoopula, spoon, or spatula to collect the soil sample and place in a stainless steel, glass, Teflon® or disposable bowl [go to Step (5)].

NOTE 2: A trier sampler may be used to collect a core of soil that is fine-grained and cohesive. This sampler also is well suited for collecting soil samples from a test pit, trench, or excavation sidewalls and bottoms (at 0^0 to 90^0 angle from horizontal) when the test pit, trench, or excavation can be safely entered/exited for sample collection or when the sampling point(s) can be safely accessed remotely, without entry using an extension rod(s) connected to the trier sampler.

CAUTION

Follow all applicable ES&H requirements, guidance, and procedures before entering a test pit, trench or excavation.

4. For soil sampling with a trier proceed as follows:

- Insert the trier approximately perpendicular to the surface of the soil and rotate (one or two times) to cut a core,

- Pull the core out of the hole slowly to minimize sample loss, and

- Place sample in a stainless steel, glass, Teflon, or disposable bowl.

Process sample (Section 3.6 Sample Processing).

5. Before leaving or filling the sampling location, use a ruler or tape measure to measure the depth from which the sample was taken and record sample number and depth in the field logbook.

6. Decontaminate the ruler or tape before use at the next sampling location.

7. Backfill sampled location to grade, if appropriate, using soil IDW generated during sample collection as specified in the WP. Use of soil generated during sample collection for backfill is only permitted when sampling has not penetrated the RGA.

8. **IF** the location is to be civil surveyed (reference WP), **THEN** label and stake location using a survey pin flag, survey stake or survey paint, and record in the field logbook.

9. Pickup and bag any plastic sheeting, trash and other disposable supplies before moving to the next sampling location.

10. Repeat Steps 1 though 10 until all soil samples have been collected at the sampling location.

11. Remove all rope, barriers, placards, etc., that were used to secure the area.

3.5.2 Manual Sampling Using an Auger

1. Use a manual auger or a manually controlled mechanical or hydraulic earth drill to advance the auger to the desired depth (reference the WP).

2. Carefully stage excess IDW (soil) from the auger bucket or solid auger flight(s) on the plastic sheeting to prevent the spread of contaminants.

NOTE 1: It may be necessary to auger down, clear and remove soil from the borehole several times to reach the desired sampling depth.

3. Attach a decontaminated auger bucket on the sampler insertion equipment and insert into the borehole.

NOTE 2: Avoid hitting the borehole wall while inserting the auger bucket to mitigate sidewall sloughing and the possibility of cross contamination.

4. Mark sampler insertion equipment for a reference point.

5. Rotate the auger to collect the sample and carefully retrieve

the full bucket.

NOTE 3: Reference WP for required VOC sampling method. Also refer to Section 3.4 General Requirements, Note 3.

6. Remove the soil sample from the auger bucket using a stainless steel, Teflon®-coated or Teflon® spoon or spatula to collect the required sample and place in a stainless steel, glass, Teflon® or a disposable bowl for mixing.

7. Process sample (Section 3.6 Sample Processing).

8. Repeat Steps 1 though 7 until all soil samples have been collected at the sampling location.

9. Backfill the borehole to grade using soil IDW generated during sample collection or other approved material as specified in the WP. Use of soil generated during sample collection for backfill is only permitted when sampling has not penetrated the RGA.

10. If the location is to be civil surveyed (reference WP), label and stake location using a survey pin flag, survey stake or survey paint, and record in the field logbook.

11. Pickup and bag any plastic sheeting, trash, and other disposable supplies before moving to the next sampling location.

12. Remove all rope, barriers, placards, etc. that were used to secure the area.

3.5.3 Sampling Using a Thin-Walled Tube Sampler

A thin-walled sampler is used to collect undisturbed sample cores. A drill/direct push rig or hand operated auger may be used to reach the top of the interval to be sampled at the location (refer to WP for requirements). The thin-walled sampler then is pushed through the bottom of the borehole to collect the sample and the core is extracted from the borehole. The undisturbed sample normally is described in accordance with BJC-ES-2303, *Borehole Logging* (PRS-ENM-2303, *Borehole Logging*) prior to soil sample collection.

1. Advance the borehole to the desired sampling depth using the method specified in the WP prior to collecting the sample.

2. Stage excess soil IDW on the plastic sheeting to prevent the spread of contaminants and/or cross contamination.

NOTE 1: It may be necessary to clear and remove soil from the borehole several times to reach the desired sampling depth.

This will be dependent on the method used to drill/penetrate to the sampling depth.

3. Assemble the thin-walled sampler and attach to sampler insertion equipment (reference WP).

4. Carefully lower the thin-walled sampler and insertion equipment to the bottom of the borehole.

NOTE 2: Avoid hitting the borehole wall while inserting the sampler to mitigate sidewall sloughing and the possibility of cross contamination.

NOTE 3: Mark manual sampler insertion equipment for a reference point.

5. Rest the dead weight of the rods and sampler on the bottom of the borehole.

6. Position a drive device on the sampling rods (reference WP).

7. Shove, twist, push, or pound the tube holder into the soil using the specified method in the WP until the tube is full or refusal is met.

8. Carefully retract sampler and sample insertion equipment from the borehole to minimize loss of soil from the thin-walled tube and disassemble.

9. Immediately cover both ends of the tube with Teflon® film or microcrystalline wax (see WP for detail) and cap if the collection tube will be used as the sample container.
   IF the tube will not be used as the sample container, **THEN** proceed to Step 12.

10. Label the top and bottom end of the tube and indicate which end is to be opened at the laboratory.

11. Use silicone tape to tape both end caps to ensure retention before shipping tube to the laboratory.

NOTE 4: Electrical tape shall never be used to tape end caps.

NOTE 5: Reference WP for required VOC sampling method. Also refer to Section 3.4 General Requirements, Note 3.

12. Process sample (Section 3.6 Sample Processing).

13. Repeat Steps 1 though 12 until all soil samples have been collected at the sampling location.

14. Backfill the borehole to grade using soil IDW generated during

sample collection or other acceptable material as specified in the WP. Use of soil generated during sample collection for backfill is only permitted when sampling has not penetrated the RGA.

15. **IF** the location is to be civil surveyed (reference WP), **THEN** label and stake location using a survey pin flag, survey stake or survey paint, and record in the field logbook.

16. Pickup and bag any plastic sheeting, trash, and other disposable supplies before moving to the next sampling location.

17. Remove all rope, barriers, placards, etc., that were used to secure the area.

3.5.4 Sampling Using a Split-Barrel or MacroCore Sampler

1. Advance the borehole to the desired sampling depth using the method specified in the WP.

2. Stage excess soil IDW on the plastic sheeting to prevent the spread of contaminants and/or cross contamination.

NOTE 1: It may be necessary to clear and remove soil from the borehole several times to reach the desired sampling depth.

3. Carefully remove the drilling or penetration device from the borehole and stage excess IDW (soil) on the plastic sheeting to prevent the spread of contaminants and/or cross contamination.

4. After reaching the sampling depth, install the split spoon or MacroCore sampler (with or without liner/insert) on the drill rods (consult WP) and reinsert into the borehole when ready to sample.

NOTE 2: Avoid hitting the borehole wall while reinserting to mitigate sidewall sloughing and the possibility of cross contamination.

5. Mark sampler insertion equipment for a reference point.

6. Position the driving device on the sample insertion equipment (consult WP).

7. Rest the dead weight of the sampler, sample insertion rod, and hammer on the bottom of the borehole.

8. Power or manually drive the sampler into the soil.

NOTE 3: When using standard penetration test equipment to drive the sampler mark the drill rods in successive 6-inch increments so

that advance of the sampler can be easily observed. Advance the split-barrel sampler the required distance (generally 18 inches) with blows from the 140-pound hammer.
Count the number of blows applied for each 6-inch increment of sampler advance and record blow count in the field logbook. Refusal generally is indicated if more than 50 blows are required to advance the sampler 6 inches.

9. Carefully retrieve and open the sampler and remove tube, if used, from the borehole. Remove disturbed material in the upper end of the sample tube using a stainless steel or Teflon®-coated spatula or spoon and immediately cover both ends of the tube with Teflon® film and cap if the collection tube will be used as the sample container. **IF** the tube will not be used as the sample container, **THEN** proceed to Step 11.

10. Use of tubes/inserts/liners for the collection of soil samples for VOC analysis is preferable (refer to WP). For VOC samples, seal, label, and ship the tube intact or use an EnCore® or equivalent sampler and associated sample container to collect the VOC samples.

NOTE 4: Reference WP for required VOC sampling method. Also refer to Section 3.4 General Requirements, Note 3.

11. Process sample in accordance with the WP and as indicated in Section 3.6, Soil Sample Processing, of this procedure.

12. Repeat Steps 1 though 11 until all soil core samples have been collected at the sampling location.

13. **IF** the location is to be civil surveyed (reference WP), **THEN** label and stake location using a survey pin flag, survey stake, or survey paint, and record in the field logbook.

14. Backfill the borehole to grade using soil IDW generated during sample collection or another acceptable material as specified in the WP. Use of soil generated during sample collection for backfill is only permitted when sampling has not penetrated the RGA.

15. **IF** the location is to be civil surveyed (reference WP), **THEN** label and stake location using a survey pin flag, survey stake or survey paint, and record in the field logbook.

16. Pickup and bag any plastic sheeting, trash, and other disposable supplies before moving to the next sampling location.

17. Remove all rope, barriers, placards, etc., that were used to secure the area.

3.6 Soil Sample Processing

The type of soil sample collected may be a discrete sample or a composite sample. Each of these sample types must be processed to ensure that a homogeneous sample is collected that is representative and meets data quality objectives given in the task-specific WP.

1. Composite the soil in a stainless-steel or glass bowl as follows unless directed otherwise by governing plans and procedures. Composite samples normally are not an acceptable means of collecting samples to be analyzed for volatile organic compounds (VOC)s, semivolatile organic compound (SVOC)s, and dissolved gases.

   - Collect soil aliquots in the same manner and of generally equal proportion.

   - Ensure aliquots are well mixed to ensure that composite sample is homogeneous.

     A stainless-steel or glass bowl and stainless-steel spoon serve well for mixing purposes. It is important that waste, soil, and sediment samples be mixed thoroughly to ensure that the sample is as representative of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

   a. The material in the sample pan is divided into quarters and each quarter is mixed individually.

   b. Two quarters are mixed together to form halves.

   c. The two halves are mixed to form a homogenous matrix.

   d. This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

2. Transfer homogenized soil samples into sample container(s), using appropriate equipment (i.e., stainless-steel or Teflon® spoon, spatula, or disposable scoop) until completely full and securely tighten lid.

3. Seal sample containers and affix custody seals in accordance with BJC-ES-2708, Chain of Custody Protocol for Environmental Sampling (Chain-of-Custody forms, Field Sample Logs, Sample Labels, and Custody Seals)..

4. Sample containers should be labeled prior to collection of the sample.

Enter any remaining information on the sample label [e.g., date and time (24-hour clock)] at the time of sample collection. See BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* (PRS-ENM-2708, *Chain-of-Custody forms, Field Sample Logs, Sample Labels, and Custody Seals*).

5. Place the sample container in a cooler with ice or blue ice to maintain a preservation temperature of 4^0C as required in the WP. Cooling normally is the only preservation method required for soil samples collected in the field.

6. Repeat Steps 1 through 5 until all soils samples are collected.

7. Complete the field logbook entries in accordance with BJC-ES-2700, *Field Logbooks* (PRS-ENM-2700, *Logbooks and Data Forms*). Logbook entries shall be made at the time of sample collection and before leaving the sampling location.

4.0 POST-PERFORMANCE ACTIONS

1. Complete COC forms in accordance with BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* (PRS-ENM-2708, *Chain-of-Custody forms, Field Sample Logs, Sample Labels, and Custody*).

2. Sample containers shall undergo radiation surveys to be performed by a RCT in accordance with applicable radcon procedures.

3. In coordination with the RCT, release the sample(s) and related COC documentation to the transportation specialist, or designee, for shipment to an off-site laboratory or deliver the sample(s) to the on-site laboratory.

4. Any sampling equipment, tools, or supplies that are contaminated as a result of their use during sample collection and not otherwise intended to be disposed of will be packaged or containerized for removal from the sampling location and decontaminated in accordance with BJC-ES-2702, *Decontamination of Sampling Equipment an Devices* (PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*). Prior to transporting contaminated equipment, tools, or supplies, contact the transportation specialist.

5. Any IDW generated during soil sampling activities shall be collected, segregated into appropriate containers, labeled, and disposed of (or treated prior to disposal) in accordance with the requirements in the WP. Prior to transporting IDW, contact the transportation specialist.

5.0 RECORDS

The field logbook, sample container COA or certificates of cleanliness, and COC Form(s) are project records and shall be maintained according

to BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

6.0 SOURCE DOCUMENTS

- U.S. Environmental Protection Agency. November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*. Region 4, Environmental Compliance Branch, Athens, GA.
- ASTM D 1452 (American Society for Testing and Materials)– 80 (Reapproved 2000). *Practice for Soil Investigation and Sampling by Auger Borings.*
- ASTM D 1587 – 00. *Method for Thin-Walled Tube Geotechnical Sampling of Soils.*
- ASTM D 2937 – 04. *Density of Soil in Place by the Drive-Cylinder Method.*
- ASTM D 3550 - 01. *Practice for Ring-Lined Barrel Sampling of Soils.*
- ASTM D 4700 – 91 (Reapproved 1998). *Soil Sampling from the Vadose Zone.*
- ASTM D 5451 – 93 (Reapproved 2004). *Standard Practice for Sampling Using a Trier Sampler.*
- ASTM D 5633 – 04. *Practice for Sampling with a Scoop.*
- ASTM D 5784 – 95. *Guide for Use of Hollow-Stem Augers for Geo-environmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices.*
- ASTM D 6169 – 98 (Reapproved 2005). *Guide for Selection of Soil and Rock Sampling Devices with Drill Rigs for Environmental Investigations.*

ATTACHMENT A
DEFINITIONS/ACRONYMS

DEFINITIONS

COMPOSITE SOIL SAMPLE – A soil sample that consists of a number of discrete soil samples collected from a body of material and homogenized prior to analysis. The objective of soil sample compositing is to represent the average condition of the sampled soil.

DISCRETE SOIL SAMPLE – A soil sample collected from one specific horizon and vertical interval (usually 6 inches).

DIRECT PUSH – Direct push sampling involves pushing a small-diameter hollow steel rod into the ground to a selected depth and extracting a soil or groundwater sample, and can be used in most materials that can be augured or sampled with a split spoon. It can be attached to cone penetrometer rods and driven into the soil with hydraulic rams.

HOLLOW-STEM AUGER DRILL RIG - The hollow-stem auger column rotates as it drills into the ground and is designed to push soil up and out of the borehole along the outside of the auger. The auger itself is driven either mechanically or by a hydraulically-powered drill rig. A plug is placed through the auger to prevent soil from rising through the hollow portion of the stem. Samples are retrieved by retracting the plug and lowering the sampler (e.g., split spoon w/ tube insert, thin-walled tube collection tube through the auger and driving or pushing the sampler to capture the sample).

MANUALLY CONTROLLED MECHANICAL OR HYDRAULIC EARTH DRILLS - A mechanically or hydraulically powered portable drilling unit whose position, orientation, location, and direction is controlled by one or more operators.

MANUALLY OPERATED AUGER – Manually operated augers consist of an auger bit, a solid or tubular drill rod, and a "T" handle. When the drill rod is threaded, extensions can be added or auger bits interchanged. The auger tip drills into the ground as the handle is rotated, and soil retained on the auger tip is brought to the surface and may used as the soil sample. Alternately, augers can be used to bore to the desired sampling depth, and a tube sampler used for collection. Diameters typically range between 2.54 and 10.16 cm (1 and 4 in.). Augers normally are used in conjunction with 0.9-1.2 m (3-4 ft) metal shafts and T-handles.

PNEUMATIC HAMMER - A pneumatic hammer (jackhammer) is a portable percussive-drill, operated by compressed air and can be used to drive samplers, install drive points, drill rock, break up pavement, etc. It works in the manner of a hammer by jabbing but not by rotating.

PUSH OR DRIVE SAMPLERS - Pushed or driven samplers penetrate into the soil without rotation and may be used for obtaining disturbed samples of most soils.

SAMPLE HANDLER - The person (or designee) responsible for ensuring that sampling activities are performed in accordance with the current, approved task-specific WP and associated procedures, and approving (or coordinating the approval of) deviations there from. Specific position titles with these responsibilities may be designated as these: project technical lead, sampling manager, lead engineer, sampling team leader, or task leader.

ATTACHMENT A
DEFINITIONS/ACRONYMS

SPLIT SPOONS - Split spoons are tubes constructed of high strength alloy steel with a tongue and groove arrangement running the length of the tube, allowing it to be split in half. The two halves are held together by a threaded drive head assembly at the top, and a hardened shoe at the bottom, with a beveled cutting tip. The sampler can be driven manually or by a 140-pound weight dropped through a 30-inch interval.

TEST PITS - Test pits are used for exposing and sampling the subsurface soil section. The test pit must be large enough to allow detailed description of the material in situ and allow soil sampling for undisturbed samples. In plan view the test pit will be square, rectangular, or circular.

TEST TRENCHES - Test trenches are used to perform a function similar to test pits, but offer the distinct advantage of a continuous exposure of the continuity and character of the subsurface soil section. Test trenches may be excavated with backhoes, trackhoes, or bulldozers depending upon the required size and depth of the trench.

THIN-WALLED SAMPLERS - The thin-walled open sampler consists of a tube affixed to a sampler head assembly, which may or may not be equipped with a check valve. Thin-walled tubes are sharpened on one end and, therefore, may be easily damaged by buckling or by blunting or tearing of the cutting edge as they are driven into stiff or coarse soils. To reduce the potential for damage, the tube should be pushed rather than driven. The basic principle of operation of the thin-walled sampler is to force the cylindrical tube into the soil in one continuous push without rotation.

THIN-WALLED TUBES - Tubes are similar to augers except that a tube with a cutting tip is attached to the drill rod. Instead of being rotated, the tube is pushed into the soil. Often, augers are used to drill the hole and tubes are used to collect the sample. Sample collection procedures are similar to split-spoon sampling except that the tube is pushed into the soil, using the weight of the drill rig, rather than driven.

TRIER - A hand-operated trier consists of a tube that has been cut in half, a solid or tubular drill rod, and a "T" handle. When the drill rod is threaded, extensions can be added for extended reach. The tip of the trier is sharpened which allows the sampler to cut into soil.

ACRONYMS

AHA - Activity Hazards Analysis
COA - Certificate of Analysis
COC - chain of custody
IDW - investigation-derived waste
PEMS - Project Environmental Measurements System database
PPE - personal protective equipment
PRS- Paducah Remediation Services, LLC
RADCON - Radiological Controls
RCT- radiological controls technician
RWP - radiological work permit
VOC- volatile organic compound
WP - work package

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Environmental Monitoring	**PRS-ENM-2700**	**REV. NO. 0**
SUBJECT MATTER AREA: Logbooks	**PREPARER: Tracy Kulik**	**Page 1 of 14**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** **Tracey Brindley (Signature on File in DCC)** **3/5/07**	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **Logbooks and Data Forms**		
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Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of PRS documentation and to establish Document Control as the control point for tracking document numbers. This document replaces BJC-ES-2700, Rev. 0, *Field Logbooks*.	All

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1.0 PURPOSE

The purpose of this procedure is to establish criteria and guidelines for content and control of data forms and logbooks and to provide guidelines for accurate and complete documentation of activities.

2.0 SCOPE

The requirements of this procedure apply to work performed for PRS and its subcontractors at the Department of Energy owned Paducah Site.

These requirements apply to all types of data forms that document data or other pertinent information (e.g., sample collection information, field measurements, and data assessment checklists) and logbooks that document an activity (e.g., site, project, sampling, laboratory, and maintenance).

The requirements in this procedure do not apply to personal or communication logbooks. Chain-of-custody and sample labels are specifically discussed in PRS-ENM-2708, *Chain-of-Custody forms, Field Sample Logs, Sample Labels, and Custody Seals.*

3.0 PRECAUTIONS AND LIMITATIONS

Logbooks are a part of the permanent project records. All logbook entries shall be factual, detailed, and objective. All logbook entries shall be made using black indelible ink. Use of correction tape and white-out are not permitted. Void entry errors by drawing a single line through the entry; and initial and date the correction. Insertions after the initial entry should be initialed and dated.

4.0 PROCEDURE

4.1 Pre-Performance Activities

- Individuals who record information in a data form or logbook shall be knowledgeable of the latest version of this procedure before beginning any activities.
- Obtain bound logbooks. Each page in the logbook shall be sequentially numbered. Logbooks include either daily logbooks or activity-specific logbooks developed using preprinted forms.

4.2 Data Form and Logbook Development

<u>Data Forms</u>

Project Manager or designee

4.2.1 Develop data forms under a controlled process by meeting the following criteria:

1. Data Forms shall be controlled and identified through a plan,

procedure, work instruction, or operator aid.

2. Data Forms shall have an identifying number on the bottom left hand corner of the form which consists of these things:

   - Controlling procedure, work instruction, or operator aid number and revision;
   - Data Form Number;
   - Date the form was developed/revised.

3. Forms shall include a date and signature of the person recording the information.

Logbooks

Field Representative

4.2.2 Record the following information, if applicable, on the outside of the front cover of each logbook using indelible ink - may be a paint pen.

- Project name and number
- Unique logbook name and number
- Document control number
- Activity or site name
- Start date of the logbook
- Completion date of the logbook (when completed)

Field Representative

4.2.3 Record the following information on the inside of the front cover of each logbook using indelible ink.

- Logbook Number
- Project Manager's name
- Return address
- Important phone numbers, as applicable
- Radio call numbers, as applicable
- Emergency contacts, as applicable

Field Representative

4.2.4 **IF** pages have not already been pre-numbered, **THEN** manually number all of the pages before the initial use of the logbook.

Field Representative

4.2.5 The first three pages of the logbook shall be reserved for a Table of Contents. Mark the first page with the following heading:

TABLE OF CONTENTS

Page(s)	Description of Activity	Date

EXAMPLE:

1-8 Sonic Drilling BRW-108 07/27/04

Field Representative

4.3 Data Form and Logbook Storage and Control

Store the data forms and logbooks in accordance with PRS-DOC-1009, *Records Management, Administrative Records, and Document Control.*

Data Forms

Maintain control of blank data forms by ensuring the form used is the current form from the current controlled procedure.

Logbooks

Logbooks shall be controlled to ensure tracking, handling, and usage so they are not lost or damaged. **IF** multiple logbooks are used for a project, **THEN** maintain a project logbook inventory.

Field Representative

4.3.1 A sequential document control number shall be assigned to each logbook.

Field Representative

4.3.2 Keep logbooks on-site unless written permission has been given in advance by a Project Manager or designee to take a logbook off-site.

Field Representative

4.3.3 The project shall maintain a logbook sign-in/out sheet to identify the individual making entries into the logbook and to provide a reference for unique signatures and initials.

- The logbook shall be signed in/out by the individual responsible for its care and custody.
- The person that signed out the logbook must maintain responsibility for a logbook until such time that either custody is transferred to another team member, or the logbook has been returned to its designated storage location upon completion of the daily activities.

Field Representative

4.3.4 **IF** the custody of the logbook is transferred, **THEN** place an entry in the logbook that documents the change in custody

- **IF** the person maintaining custody of the logbook is present, and is being aided by a second person, **THEN** transfer of custody is NOT required.
- Record full name of the person releasing custody and date in the logbook
- Record full name of the person receiving custody and date in the logbook.

Field Representative

4.4 Data Form and Logbook Entry Requirements

Data Forms

See attachment B for example Data Form entries.

The use of correction tape or white-out is not permitted.

1. Entries shall be made in black, indelible ink, as legibly as possible.
2. Place each entry on blank lines provided on the data form.
3. Indicate any deletions or changes by marking through the original material with a single line; then initial and date the change. No obliteration of incorrect entries, including correction tape or fluid, shall be made.
4. Any error on a data form or logbook entry should be corrected by the same person who made the original entry, whenever feasible.
5. Indicate any insertion by initialing and dating the insertion.
6. **IF** any blanks within the data entries on a form are NOT used, **THEN** write "N/A" (not applicable) in the blank line or empty space.
7. Identify each unused section by filling in the unused area with a "Z" line, initials, and date.

Logbooks

See attachment C for example Logbook entries.

The intent of keeping a logbook is for the reader to be able to recreate an event as accurately and completely as possible based on the written information. A logbook is a factual, chronological record of the activities throughout the day. The logbook author must include observations and descriptive notations. However, the author must take care to be objective and record no opinions or subjective comments.

DO NOT remove any pages for any reason. The use of correction tape or white-out is not permitted.

Record the information directly into the logbook, DO NOT record notes elsewhere for recopying of the information into the logbook at a later time.

1. Entries shall made in black, indelible ink, as legibly as possible, and entered on consecutive lines and pages.
2. Indicate any deletions or changes by marking through the original material with a single line; then initial and date the change. No

Field Representative

obliteration of incorrect entries, including correction tape or fluid, shall be made.

3. Any error on a data form or logbook entry should be corrected by the same person who made the original entry, whenever feasible.

4. Indicate any insertion by initialing and dating the insertion.

5. Precede each entry with the time (24-hour clock).

6. DO NOT skip lines. There shall be no blank spaces or pages between entries.

7. Start each day in the logbook on a right hand page.

8. Record the following information each day on the first page of the section of the logbook to be used for the task:

   - Task to be Performed
   - Location Worked
   - Project Lead's Name

9. Document important relevant correspondence (written, personal, and telephone conversations)

10. Record the applicable information during field activities, such as:

    a) Date (month/day/year) at the top and bottom of each page.

    b) A brief description of the activities that will be conducted during the field event.

    c) Names of personnel assigned to the task, including subcontractors.

    d) A list of all field equipment that will be used and identifying or serial numbers.

    e) Description of present weather conditions (e.g., temperature, precipitation and form, cloud cover, wind direction, and velocity).

    f) Site conditions (upon arrival, changes during the field activities, and upon departure) that may affect work performance or impact sample quality (e.g., weather conditions, paint odor, vehicle or equipment exhaust upwind of sampling location, discoloration of ground or water surface around sampling point).

    g) Include descriptions of any general problems encountered and resolutions found.

    h) Names, affiliations, and times of arrival and departure of all visitors or observers to the work site. If unsure, ask and record the purpose of their visit.

Field Representative

i) Start and stop times (24-hour clock) of field activities. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged.

j) Any deviations or one-time difficulties encountered in the field activities in sufficient detail to completely describe the activity or event.

k) Describe any issues, concerns, or problems and their resolution, including any equipment failures with a description of downtime, standby time, repairs, replacements, and/or recalibrations, as applicable.

l) Decontamination activities (if any), noting procedures used or reference to a project-specific plan specifying decontamination practices used.

m) Field measurements taken using instruments, which may include the following:

- Name of technician;
- Instrument(s) utilized;
- Calculations, results, and calibration data for measurement equipment;
- Media that were measured;
- Type of measurement (e.g., temperature, turbidity, conductivity, pH);
- Sample identification number from which the measurement was taken;
- Time of sample collection;
- Measurement results with appropriate units of measure; and
- Any special comments/observations.

n) Results of health and safety monitoring, including instrument make, model, serial number, and calibration standards used.

11. At the end of all entries for each day, or at the end of a particular event, if appropriate, a “Z” line shall be drawn through any blank space remaining at the bottom of each page and shall be initialled and dated by the individual making the entry.

12. Identify each unused logbook page by filling in the unused area with a “Z” line, signature, and a date.

13. Sign and date the bottom of each page. **IF** a “Z” line and

signature and date are used to void a blank page, **THEN** an additional signature and date at the bottom of the page are not required.

4.5 Requirements for Data Forms Placed in a Logbook

Field Representative

4.5.1 Labels and stickers never should be placed over previously recorded information.

Field Representative

4.5.2 Place a signature across the corner of the label/sticker onto the logbook page to signify that the label/sticker should be considered part of the page.

Field Representative

4.5.3 Make entries on the data form as required in Section 4.4.

Field Representative

4.5.4 **IF** additional information is necessary to provide pertinent information and no additional space is available on the data form, **THEN**

record the additional information under the data form, if space is available, or go to the next available place in the logbook to record the additional information.

- If the additional information is not on the same page as the data form, indicate on the data form the page number of the additional information.
- If the additional information is not on the same page as the data form, within the entry, indicate the page number of the data form associated with the additional information.

4.6 Data Form and Logbook Reviews

Project Manager or Designee

4.6.1 Conduct periodic reviews, at least monthly during field operations, of data forms and logbooks (including data forms placed in logbooks) to verify the following:

- Accuracy of entries;
- Legibility and clarity of entries;
- Completeness to ensure that at least the minimum required information is recorded;
- Consistency of information recorded;
- Signature and date of entries by the designated team member;
- Compliance to the requirements in this procedure.

Project Manager or Designee

4.6.2 Notify appropriate personnel of corrections or clarifications that may be needed. **IF** any discrepancies or deviations from this procedure are found, **THEN**

the reviewer shall inform the project manager as soon as practicable and obtain resolution before signing the logbook page.

Project Manager or Designee

4.6.3 When a logbook or data form review has been completed, the reviewer shall sign and date the bottom right-hand corner of the logbook or data form page.

4.7 Post-Performance Activities

Project Manager or Designee

Submit the original logbooks and data forms to the Document Management Center upon completion of the task or when the logbook is full, which ever occurs first.

5.0 RECORDS

Data forms, logbooks, and logbook sign in/out sheets are project records and shall be processed and maintained according to PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*.

6.0 SOURCE DOCUMENTS

U.S. Environmental Protection Agency, November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, Section 3.5. Region 4, Environmental Compliance Branch, Athens, GA.

Attachment A
DEFINITIONS/ACRONYMS

DEFINITIONS

Data Forms - A loose, unbound, uniquely identified, single-sided or double-sided page(s) usually with pre-printed table for convenient, manual data entry. Data forms may be printed on sticker paper to be placed in a logbook. Forms are used for the permanent recording of information pertaining to various data collection activities (e.g., sample collection information forms, field measurement forms, data assessment checklists, etc.).

Logbook - A bound book with sequentially numbered pages used to create a permanent, near real-time record of activities and conditions, significant events, observations, and measurements that occur during each day of field activities.

Project Manager - The person (or designee) responsible for ensuring that sampling activities are performed in accordance with the current, approved plans or other governing documents and associated procedures. The project manager approves (or coordinates the approval of) deviations from the approved plans or other governing documents.

Reviewer - A person who is knowledgeable about, but independent from, the tasks being performed and is responsible for verifying conformance to procedural requirements.

ACRONYMS

PRS - Paducah Remediation Services, LLC

Attachment B
EXAMPLE OF COMPLETED DATA FORM
Page 1 of 1

PROJECT:	EM	LOCATION:	MW102	DATE:	6/15/2006
FREQUENCY:	Quarterly	ARRIVAL TIME:	1230	DEPARTURE TIME:	1304
SAMPLE NO:	MW102B-00	SAMPLED BY:	(initials)	SAMPLE TIME:	1255
WELL DEPTH:	95 feet	WATER DEPTH:	65 feet	BAROMETER:	30.15 in/Hg
PURGE START:	1240	PURGE STOP:	1250	PURGE AMOUNT:	35 gallons
WEATHER:	☐ sunny ☒ cloudy ☐ rainy ☐ snowy			TEMPERATURE:	75 °F

FIELD MEASUREMENTS

TIME	D. O. (mg/L)	TEMPERATURE (°C)	pH	CONDUCTIVITY (mhos/cm) TCK 6-15-06
1252	9.1	14.7	7.4	~~335~~ 235
N/A	N/A	N/A	N/A	N/A
↓	↓	↓	↓	↓

EQUIPMENT DECONTAMINATION

DESCRIPTION: Deconned sample tubing using procedure PRS-ENM-2702

TIME: 1300

COMMENTS/OBSERVATIONS: While the sample was being collected a large truck drove by and strong exhaust fumes were noticed. TCK 6-15-06

TCK 6-15-06

SIGNATURE: *Tracy Kulik* DATE: 6-15-06

Attachment C
EXAMPLE OF COMPLETED DATA FORM
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Tracy Kulik 6-15-06

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C-400 RDSI Thursday 6/15/06

DAILY SUMMARY

Task: Soil boring

Location: C-400 building

Project Lead: Mike Clark

Team Members: name, role, company
name, role, company

Equipment: Geoprobe SN: 123XYZ
augers, split spoon samplers
Hydac pH, meter #2

Weather: cloudy, 75 °F, light wind from the south

Visitors: Jim Johnson, EPA, 9:00-9:15
Julie Smith, EPA, 9:00-9:15

Issues: Geoprobe broke down. Had to be taken offsite for repairs at 1300. Shut down for the day.

Sampled by procedure PRS-ENM-1234

Decontaminated equipment by procedure PRS-ENM-2702.

TCK 6-15-06

Tracy Kulik 6-15-06

Attachment C
EXAMPLE OF COMPLETED DATA FORM
Page 2 of 2

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C-400 RDSI Thursday 6/15/06

0630	Arrive on site and begin setting up at soil boring 001
0940	Start drilling soil boring 001
0648	Collect soil sample SB001-00-05
0700	Collect soil sample SB001-05-10
0708	Collect soil sample SB001-10-15, sweet odor was noticed coming from soil before the sample was placed in the jar.
0710	ES&H performed monitoring of the area and detected 50 ppm VOCs. Project manager was contacted and PPE was increased from level D to level B. See ES&H logbook for more info.
0730	Re-start drilling efforts
0740	Collect sample soil SB001-15-20
0750	Completed sampling activities at soil boring 001.
0755	Decontaminate sampling and drilling equipment.
0845	Collect ground water sample at MW-12 see page 22 for details on parameters.
0859	Begin setting up at soil boring 002
0900	Jim Johnson and Julie Smith arrive from DOE. They talked with Mike Clark.
0905	Start drilling soil boring 002
0915	Collect soil sample SB002-00-05
0921	Collect soil sample SB002-05-10
0930	Collect soil sample SB002-10-15

Tracy Kulik 6-15-06

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C-400 RDSI Thursday 6/15/06

0942	Collect soil sample SB002-15-20
1000	Completed sampling activities at soil boring 002.
1010	Decontaminate sampling and drilling equipment and break for lunch.
1145	Begin setting up at soil boring 003
1155	Start drilling soil boring 003
1201	Collect soil sample SB003-00-05
1215	Collect soil sample SB003-05-10
1218	Hit refusal and decide to offset the boring.
1230	Begin setting up at offset soil boring 003 approximately 12 in south of the original location
1245	Begin drilling offset boring 003
1250	At approximately 9 feet BGS, hit refusal. A second attempt was made to continue down, and the Geoprobe broke down.
1300	Geoprobe will require extensive repairs and will need to be taken offsite.
1310	Decontaminate sampling and drilling equipment and stop for the day.

TCK 6-15-06

Tracy Kulik 6-15-06

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OWNER: Environmental Monitoring	**PRS-ENM-2702**	**REV. NO. 0**
SUBJECT MATTER AREA: Engineering	**PREPARER: K. Holland**	**Page 1 of 13**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** Tracey Brindley (Signature of File in DCC) 3/5/07	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY:**		
TITLE: **DECONTAMINATION OF SAMPLING EQUIPMENT AND DEVICES**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 3/12/07**	
USQD/UCD No: USQD-PH-SITE-0037/R1-UCD-PH-SITE-0075/R1	**REQUIRED REVIEW DATE: 3/12/10**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

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1.0 PURPOSE

This procedure establishes methodologies for cleaning and decontaminating sampling equipment and devices that come into contact with sample media and/or contaminants.

The objectives of decontamination are to remove contaminants from surfaces, mitigate the spread of contaminants to other uncontaminated surfaces, prevent cross-contamination of sample matrices, and to minimize personnel exposure and waste volume.

2.0 SCOPE

This procedure shall be used for decontamination of sampling equipment and devices used for characterization and cleanup verification activities performed by PRS personnel. This procedure is not intended to provide direction on the decontamination of large field equipment and equipment components (e.g., backhoes, fork lifts, trackhoes, front loaders, graders, well drilling equipment, drill bits, drilling rods, etc.). For those decontamination activities, see BJC-ES-2701, *Large Equipment Decontamination* (PRS-FCD-2701, *Large Equipment Decontamination*).

3.0 PRECAUTIONS AND LIMITATIONS

Refer to applicable project AHAs for precautions concerning specific hazards that may be encountered and to determine the required PPE.

Additional cleaning and decontamination procedures and methods may be required because of differing contaminant characteristics. Additional requirements for decontamination should be stated in the task-specific WP or governing documents.

4.0 PROCEDURE

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by Paducah Remediation Services, LLC.

4.1 Quality Assurance/Quality Control

4.1.1 QC Samples

The number or frequency of equipment rinsate samples shall be collected as specified in the task-specific WP and prepared in accordance with BJC-ES-2704, *Trip, Equipment and Field Blank Preparation* (PRS-ENM-2704, *Trip, Equipment and Field Blank Preparation*).

4.1.2 Control and Documentation of Deviations

Any departures from the requirements specified in the approved, task-specific WP or this procedure shall be authorized through the Project Manager or designee. Prior to deviating from those requirements, authorization and WP revision shall be in accordance with BJC-FS-1001, *Work Control Process* (PRS-WCE-0018*, Paducah Environmental Remediation Project Work Management Program*). Any deviations and one-time difficulties encountered in the field concerning sample collection or related activities shall be documented in the Field Logbook by the Sample Handler.

4.2 Field Preparations

1. Assemble the necessary equipment, tools, and supplies (see Section 4.3) to ensure that sufficient materials are available for decontamination activities.

2. In accordance with SH-A-5181, *Hazardous Materials Information System* (PRS-ESH-5181, *Hazardous Materials Information* System) and BJC-EH-5140, *Hazard Communication* (PRS-ESH-5140, *Hazard Communication*) MSDS for solvents that will be used for cleaning and decontamination shall be submitted to the PRS HMIS Coordinator at least five (5) working days prior to the chemicals being brought on site.

3. All appropriate MSDS shall be available at the sampling location.

4. Cleaning materials shall be stored as follows until used:

   - Soap shall be kept in clean plastic, metal, or glass containers.

   - Solvents shall be stored in the closed original containers. Solvents may be applied using a low pressure nitrogen system fitted with a Teflon® nozzle or by using Teflon® squeeze bottles.

   - Tap water may be kept in clean tanks, hand pump sprayers, squeeze bottles, or applied directly from a hose.

   - Analyte-free water shall be stored in clean glass, stainless steel, or plastic containers that can be closed when not being used. Analyte-free water may be applied from Teflon® squeeze bottles.

   - Organic-/analyte-free water must be stored in clean glass, Teflon®, or stainless steel containers prior to use. It may be applied using Teflon® squeeze bottles.

- Nitric acid shall be kept in the glass container it is received in and placed in squeeze bottles prior to application.

NOTE: Hand pump sprayers (including stainless steel sprayers) generally are not acceptable as storage or application containers for the above materials (except tap water) due to gaskets and seals that may contaminate the solutions.

4.3 Equipment, Tools, and Supplies

The sampling supervisor (or designee) is accountable for gathering all needed equipment, tools, and supplies to safely and successfully complete the cleaning and decontamination activities. The items listed in this section may be used as a guide, but may not represent a complete list. Refer to the task-specific AHA(s) and WP, and the RWP (if one is required), to determine what supplies, materials and equipment are needed to safely execute the decontamination activities.

1. Soap (phosphate-free laboratory detergent such as Liquinox®)
2. Solvent, as applicable
3. Tap water
4. Analyte-free (deionized) water
5. Organic-/analyte-free water
6. Nitric acid, as applicable
7. Steam cleaner or high pressure hot water washer capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam ≥ 200 ^{o}F, with a soap compartment
8. Natural bristle brushes
9. Buckets
10. Aluminum foil, plastic wrap, zipper-type plastic bags, and clean plastic trash-size bags
11. Plastic sheeting or other impermeable liner for decontamination area
12. Paper towels, clean cloths, or rags
13. Spill control kit
14. Absorbent pads
15. PPE and other safety equipment specified by the AHA and RWP
16. MSDS, as applicable

17. Field Logbook

18. Indelible marking pens

The sampling team members as assigned by the sampling supervisor shall complete the following steps.

NOTE 1: Nondisposable sampling equipment and devices shall be decontaminated prior to reuse as described under this section. Disposable sampling equipment and devices shall be handled as IDW in accordance with the requirements in the approved, task-specific WP.

NOTE 2: Record all decontamination activities in the Field Logbook in accordance with BJC-ES-2700, *Field Logbooks* (PRS-ENM-2700, *Logbooks and Data Forms*)

4.4 Segregation of Sampling Equipment and Devices

1. Sampling equipment and devices that need to be decontaminated shall be stored separately from new or cleaned (decontaminated) sampling equipment and devices.

2. After decontamination, sampling equipment and devices shall be handled only when wearing clean, chemical-resistant gloves to prevent re-contamination.

3. Sampling equipment and devices that have been surveyed by a RCT after decontamination, found to meet the criteria in EH-4516, *Radioactive Contamination Control and Monitoring* (PRS-RAD-1109, *Radioactive Contamination Control and Monitoring*), and that will not be reused immediately in a Radiological Area, Soil Contamination Area, or Radiological Buffer Area shall be green-tagged in accordance with EH-4517, *Posting and Labeling* (PRS-RAD-1108, *Posting and Labeling*). Such sampling equipment and devices also may be wrapped in aluminum foil or plastic (put larger devices such as pumps or hoses into clean, trash-size plastic bags), as appropriate, and green-tagged as clean, marking on the plastic or foil, or segregating into designated bins, and noting the date of decontamination.

4.5 Temporary Decontamination Area

NOTE: Decontamination of sampling equipment and devices shall be conducted at the designated decontamination area for the facility, if available, or at a temporary decontamination area preferably located close to the work site so that contaminants are not spread or transferred to other equipment.

1. **IF** it is necessary to construct a temporary decontamination area, **THEN** determine whether the sampling equipment and devices are expected to be radiologically and/or chemically contaminated.

a. For Known or Potential Radiological Contamination – it is preferable that the decontamination area be constructed of clean material in an area that already is posted as a Contaminated Area. However, to the extent possible, select a location that previously had little to no surface contamination.

NOTE 1: This approach avoids contamination of a previously uncontaminated area.

NOTE 2: **IF** the temporary decontamination area is constructed in a clean, currently un-posted area, **THEN** the area shall be screened by an RCT and posted in accordance with the requirements in EH-4517, *Posting and Labeling* (PRS-RAD-1108, *Posting and Labeling*).

b. For Known or Potential Chemical Contamination only– construct the decontamination area in an area known or believed to be free of surface contamination.

2. If possible, set up a temporary decontamination area on a generally level surface that facilitates the capture, removal and control of wastewater and precipitation (if any), by laying down impermeable plastic sheeting with no seams, rips, or tears. Ensure that the temporary decontamination area is capable of containing all decontamination fluids.

NOTE 3: Depending on expected weather conditions, the temporary decontamination area should be kept covered when not in use to prevent the accumulation of precipitation.

3. Sawhorses or racks constructed to hold sampling equipment or devices while being decontaminated should be high enough above the floor of the decontamination area (i.e., at least two feet) to prevent equipment from being splashed.

4. Rinse water should be collected off the plastic sheeting frequently during the decontamination process as a safety precaution to help prevent slipping and to mitigate the spread of contaminants. Wastewater shall be handled as IDW.

NOTE 4: Solvent and 10% nitric acid rinsates shall be collected in separate containers for treatment or proper disposal as IDW. No solvent or nitric acid rinsates shall be placed in the temporary decontamination area.

5. At the completion of site activities, restore the area by removing the plastic sheeting after all waste, IDW water, and precipitation (if any), has been removed, properly containerized and labeled for treatment or disposal.

4.6 General Sampling Equipment and Device Decontamination

NOTE 1: If potentially radiologically contaminated sampling equipment and devices are to be removed from a Radiological Area for decontamination, the sampling equipment and devices shall first be surveyed by an RCT to determine removable and/or fixed surface contamination levels for comparison to the criteria in EH-4516, *Radioactive Contamination Control and Monitoring* (PRS-RAD-1109, *Radioactive Contamination Control and Monitoring*). Surveyed sampling equipment and devices shall be tagged in accordance with EH-4517, *Posting and Labeling* (PRS-RAD-1108, *Posting and Labeling*), prior to being re-located. Release of surveyed sampling equipment and devices to other areas shall be approved by the RCT or PHP, as appropriate, in accordance with EH-4516, *Radioactive Contamination Control and Monitoring* (PRS-RAD-1109, *Radioactive Contamination Control and Monitoring*).

NOTE 2: Before selecting a cleaning method or decontamination approach for specific field test equipment or instrumentation (e.g., pH meters, thermometers, dissolved oxygen meters), refer to the manufacturer's instruction manual for any recommendations and precautions in order to avoid the possibility of damage to instrument components.

1. Clean with tap water and soap using a brush to remove particulate matter and surface films.

NOTE 3: If sampling equipment or devices were used to collect samples that contain oil, grease or other hard to remove materials, then it may be necessary to rinse the equipment or device several times with pesticide-grade acetone, hexane, or petroleum ether to remove those materials before proceeding with the initial cleaning step.

2. If appropriate and necessary, sampling equipment and devices may be cleaned with soap and high pressure steam or hot water as an alternative to brushing. Sampling equipment and devices that are cleaned in this manner should be placed on racks or saw horses at the decontamination area.

NOTE 4: PVC or plastic items shall not be steam or hot water pressure cleaned.

3. Rinse sampling equipment or devices thoroughly with tap water.

4. If required by the task-specific WP, rinse Teflon® and glass sampling equipment and devices with a 10% nitric acid solution.

5. Rinse sampling equipment and devices thoroughly with analyte-free water.

6. If required by the task-specific WP (e.g., when collected samples will undergo trace organic or inorganic constituent analyses), rinse sampling equipment and devices thoroughly with an appropriate solvent.

NOTE 5: Do not solvent rinse PVC or plastic items.

7. **IF** a solvent-rinse is required, **THEN** follow with a thorough rinse using organic-/analyte-free water and air dry.

NOTE 6: Do not apply a final rinse with analyte-free water following a rinse with organic-/analyte-free water.

8. If organic-/analyte-free water is not available, sampling equipment and devices should be allowed to completely air dry.

9. Tag, label, and store decontaminated sampling equipment and devices as stated in Section 4.4.

4.7 Well Sounder or Tape Decontamination

1. Wash with soap and tap water.

NOTE: Only that portion of the well sounder cable or tape that has a potential to have come into contact with contamination needs to be cleaned. In some cases, these units may have several hundred feet of cable or tape that otherwise would not necessarily have to be cleaned.

2. Rinse with tap water.

3. Rinse with analyte-free water and air dry.

4. Tag, label, and store decontaminated equipment as stated in Section 4.4.

4.8 Pump Decontamination

WARNING

In order to avoid the possibility of electrical shock, make sure that the pump is unplugged during cleaning and decontamination.

CAUTION

In order to avoid damage to pumps or other devices operated by a controller, make sure that the controller is unplugged and does not become wet during cleaning and decontamination activities.

1. Pump soapy water through the hose to flush out any residual purge water.

2. Using a brush or wipe, scrub the exterior of the contaminated hose, pump, and electrical cord with soap and tap water.

3. Rinse the soap from the outside of the hose, pump, and electrical cord with tap water.

4. Rinse the hose with analyte-free water.

5. Pump tap water through the hose to flush out soapy water (approximately 1 gallon).

6. Pump analyte-free water through the hose to flush out the tap water. **IF** the pump has a reverse mode, **THEN** purge with the pump in reverse mode.

7. Rinse the tap water residue from the outside of hose, pump, and electrical cord with analyte-free water (approximately ¼ gallon) and let dry.

8. Tag, label, and store decontaminated equipment as stated in Section 4.4.

4.9 PCB Contaminated Sampling Equipment

PCB contaminated sampling equipment must be decontaminated and verified clean according to TSCA specifications. Any equipment to be reused that was suspected to have come in contact with an M_L labeled PCB material and/or a PCB source ≥ 50 ppm is considered to be PCB CSE. **Do not leave PCB CSE unattended.**

4.9.1 Following the sampling event, all PCB CSE must be:

- Wiped free of loose liquids and materials at the sampling point
- Double-wrapped in plastic bags with an M_L label affixed and then transported to the decontamination location.
- Decontaminated by swabbing with 100% penetone or hexane with dampened rags. The production of free liquid waste should be avoided.

4.9.2 Once decontamination operations are performed, the equipment

must be double-wrapped in plastic with M_L label and stored in a cabinet labeled PCB Dedicated Equipment Storage Cabinet

4.9.3 To reuse the sampling equipment without verification sampling, sufficient data should be generated to show that a TSCA decontamination procedure is satisfactory to clean the equipment to non-detectable levels. PCB Dedicated Sampling Equipment can be verified clean by discovery sampling.

- Obtain a 100-cm^2-wipe sample from an area on the equipment that was most likely contaminated
- Submit sample to the laboratory for analysis.

4.9.4 If the result for the 100 cm^2 wipe is <10 µg/wipe, the equipment can be deemed non-TSCA and the equipment can be allowed to be cleaned up for quality assurance concerns.

4.9.5 If the result for 100-cm^2-wipe sample is ≥ 10 µg/wipe, the PCB CSE should be disposed of as PCB waste.

4.9.6 All waste produced from the decontamination operations is considered PCB waste and must be disposed of accordingly. Contact the waste management group for guidance on handling PCB waste.

4.10 Post-Performance Actions

1. Equipment to be released off-site shall undergo radiation surveys to be performed by an RCT in accordance with EH-4515, *Radiation Surveys* (PRS-RAD-1110, *Radiation Surveys*) and EH-4516, *Radioactive Contamination Control and Monitoring* (PRS-RAD-1109, *Radioactive Contamination Control and Monitoring*).

2. Any IDW generated by the decontamination process will be collected, segregated into appropriate containers, labeled, and disposed of (or treated prior to disposal) in accordance with the requirements in the WP. Prior to transporting IDW, contact the Transportation Specialist.

5.0 RECORDS

The Field Logbook is a project record and shall be maintained according to BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

6.0 SOURCE REQUIREMENTS

- US Army Corps of Engineers, February 2001. EM 200-1-3, *Requirements for the Preparation of Sampling and Analysis Plans*.

- U.S. Environmental Protection Agency, November 2001. *Environmental Investigations Standard Operating Procedures and*

Quality Assurance Manual. Region 4, Environmental Compliance Branch, Athens, GA., Appendices B and C.

ATTACHMENT A
DEFINITIONS/ACRONYMS

DEFINITIONS

Analyte-Free (Deionized) Water: Tap water treated by passing through a standard deionizing resin column. It should contain no detectable heavy metals or other inorganic compounds at or above method detection limits as defined by a standard inductively coupled Argon Plasma Spectrophotometer (or equivalent) scan.

Equipment Rinsate Blank: A sample of analyte-free water poured over and/or through decontaminated sampling equipment. The purpose of the equipment rinsate blank is to assess the adequacy of the decontamination process.

Nitric Acid (10%) Solution: A solution made from reagent-grade nitric acid and deionized water.

Organic-/Analyte-Free Water: Tap water treated with activated carbon and deionizing units. At a minimum, it must meet the analytical criteria of analyte-free water and should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the U.S. Environmental Protection Agency Region 4 laboratory for a given set of analyses.

PCB CSE - Any equipment to be reused that is suspected to have come in contact with an M_L labeled PCB material and/or a PCB source 2 to 50 ppm.

Sampling Handler: The person (or designee) responsible for ensuring that decontamination activities are performed in accordance with the current, approved task-specific WP and associated procedures, and approving (or coordinating the approval of) deviations. Specific position titles with these responsibilities may be designated as: project technical lead, sampling manager, lead engineer, sampling team leader, or task leader.

Solvent: Pesticide-grade isopropanol. Use of a solvent other than pesticide-grade isopropanol (e.g., pesticide-grade acetone, hexane, petroleum ether) for sampling equipment cleaning and decontamination purposes must be justified in the WP.

Tap Water: Water from any municipal water treatment system. Untreated potable water is not an acceptable substitute for tap water.

ATTACHMENT A
DEFINITIONS/ACRONYMS

ACRONYMS

AHA – Activity Hazard Assessment
BJC – Bechtel Jacobs Company, LLC
CSE – Contaminated Sampling Equipment
HMIS – Hazardous Materials Information System
IDW – investigation-derived waste
MSDS – material safety data sheet
PCB – Polychlorinated biphenyl
pH – potential of hydrogen
PHP – project health physicist
PPE – personal protective equipment
PRS – Paducah Remediation Services, LLC
PSI – pounds per square inch
PVC – polyvinyl chloride
QC – quality control
RCT – Radiological Controls technician
RWP – radiological work permit
TSCA – Toxic Substances Control Act
USQD – Unreviewed Safety Question Determination
WP – work package

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Remediation Services
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OWNER: Environmental Monitoring	**PRS-ENM-2704**	**REV. NO. 0**
SUBJECT MATTER AREA: Engineering	**PREPARER: K. Holland**	**Page 1 of 8**
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PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **TRIP, EQUIPMENT AND FIELD BLANK PREPARATION**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 3/12/07**	
USQD/UCD No: USQD-PH-SITE-0036/R1-UCD-PH-SITE-0075/R1	**REQUIRED REVIEW DATE: 3/12/10**	
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0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of PRS documentation. This document replaces BJC-ES-2704, *Trip, Equipment and Field Blank Preparation*, Rev. 0; GEO-TEC-009, *Collection of Field QC Samples*, Rev. 2; and W-156-PWOS, *Collection of Field Quality Control Samples*, Rev. 1.	All

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1.0 PURPOSE

This procedure establishes guidelines for preparation and use of QC samples (Equipment Rinsate Blank, Field Blank, Trip Blank, and Field Duplicate Samples) collected during field sampling activities.

2.0 SCOPE

This procedure shall be used for environmental sampling performed by PRS personnel.

3.0 PROCEDURE

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by Paducah Remediation Services, LLC.

3.1 Equipment and Materials

The Sample Handler (or designee) is accountable for gathering all needed equipment, tools, and supplies to safely and successfully collect QC samples. The items listed in this section may be used as a guide, but may <u>not be a complete list</u>. Refer to the task-specific AHAs, WP, and the RWP (if one is required) to determine what supplies, materials, and equipment are needed to collect QC samples.

1. Analyte-free water for inorganic and radiologic analysis, if applicable.

2. Organic-free water for organic analysis, if applicable.

3. Sample containers appropriate for the samples to be collected with COAs, or certificates of cleanliness.

4. Field Logbook(s).

5. PPE and other safety equipment specified by the AHA and RWP.

6. Labels and appropriate forms for sample shipment (i.e., COC forms, custody seals, sample labels generated from PEMS data base, etc.)

7. Black indelible ink pens.

8. Plastic sheeting and Ziploc bags.

9. Decontamination equipment and supplies.

10. Cooler and sealing tape.

3.2 Summary of Method

Field QC samples are used to determine the presence and concentration of contaminants resulting from field activities and to measure/control variables in sample handling. Field QC samples may consist of field blanks, equipment rinsate blanks, trip blanks, and/or field duplicate samples.

Field QC samples assist in ensuring that the accuracy of analytical results are stated with a high level of confidence.

The frequency that field QC samples are to be collected and their appropriate preservation requirements will be defined by the task-specific WP.

3.3 Preparing an Equipment Rinsate Blank

Equipment rinsate blanks are prepared in the field using analyte-free or organic-free water as required by the task-specific WP. These samples are used to determine if contaminants have been introduced by contact of the sample medium with contaminated sampling equipment. This serves as a QC check on the cleanliness of the sampling device and, therefore, the equipment decontamination process. Equipment rinsate blanks are required only when non-disposable or non-dedicated sampling equipment is being used.

Peristaltic Pump

Pump analyte-free or organic-free water directly from the analyte-free water bottle, through the decontaminated tubing, and into the sample bottles.

Submersible Pump

Pour analyte-free or organic-free water into a clean container and pump water from the container through the decontaminated hose and into the sample bottles.

Bladder Pump

Fill a 3-m (10-ft) length of at least 10-cm (4-in.)-diameter clean pipe (sealed at one end), or other container specified in the WP, with analyte-free or organic-free water. Pump the water through the pump and into the sample bottles. It may be necessary to add water to the pipe during pumping.

Sampling Equipment and Devices

Run analyte-free or organic-free water over or through the decontaminated sample collection equipment or device and into the sample bottles.

3.4 Preparing a Field Blank

Field Blanks are prepared in the field using analyte-free or organic-free water as required by the task-specific WP. These samples are used to evaluate the potential for contamination of a sample by ambient site contaminants from a source not associated with the sample collected (e.g., air-borne fugitive dust or organic vapors). Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

1. Transport analyte-free or organic-free water to the field in a sealed container.

2. Open the container in the field near the sampling location and pour the analyte-free or organic-free water into the sample bottle.

3.5 Preparing a Field Duplicate Sample

Field duplicate samples may be either subsamples (replicate) or co-located samples and shall be collected using the same procedural requirements as a nonduplicate sample. The type of duplicate sample will be specified in the WP.

Disturbance or mixing of samples for VOC or mercury analysis must be avoided. The duplicate sample may need to be collected as a co-located sample (e.g., soil from adjacent locations or depths) to prevent the potential loss of volatile compounds/analytes.

1. Collect sample material using the same procedural requirements as a nonduplicate sample.

2. **IF** the duplicate is a subsample (replicate), **THEN** collect ample volume in order to meet the volume needed to fill two sets of bottles for all analyses being conducted and thoroughly homogenize each sample.

3. Place samples in separate, but identical containers for analysis at the same laboratories.

3.6 Preparing a Field Split Sample

Field split samples are a variation of field duplicate samples. Two or more representative portions are taken from a sample and are analyzed by different laboratories. Prior to splitting, a sample is mixed (except volatiles) to minimize sample heterogeneity.

Disturbance or mixing of samples for VOC or mercury analysis must be avoided. The split sample may need to be collected as a co-located sample (e.g., soil from adjacent locations or depths) to prevent the potential loss of volatile compounds/analytes.

1. Collect sample material using the same procedural requirements as a nonduplicate sample. Collect ample volume in order to meet

the volume needed to fill two sets of bottles for all analyses being conducted.

2. Thoroughly homogenize the sample (except volatiles).

3. Place samples in separate, but identical containers for analysis at the different laboratories.

3.7 Preparing a Trip Blank

Trip blanks are used to determine if samples were contaminated during storage and transportation to the laboratory. Trip blanks normally are used when samples are to be analyzed for VOC or tritium. Unless specified otherwise in the site-specific sampling plan, aqueous trip blanks are used for both water and soil/sediment samples.

1. Place the trip blank consisting of 2-3 pre-prepared sample vials of organic-free water in the cooler that will contain VOC samples. The trip blanks shall be kept with the investigative samples they represent from the field to the laboratory and shall be left unopened.

2. Trip blanks shall be packed for shipment with the investigative VOC samples and sent for analysis.

3.8 Post-Performance Actions

1. Post-performance activities of QC samples will be in accordance with the procedural requirements of the appropriate sampling method (e.g., surface water sampling, groundwater sampling, soil sampling) or WP requirements.

2. Complete COC forms in accordance with BJC-ES-2708, *Chain of Custody Protocol for Environmental Sampling* (PRS-ENM-2708, *Chain of Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*).

3. Sample containers shall undergo radiation surveys to be performed by a RCT in accordance with EH-4516, *Radioactive Contamination Control and Monitoring* (PRS-RAD-1109, *Radioactive Contamination Control and Monitoring*) and, as appropriate, tagged and labeled in accordance with EH-4517, *Posting and Labeling* (PRS-RAD-1108, *Posting and Labeling*).

4. If off-site laboratory analysis is required, in coordination with the RCT, release the sample(s) and related COC documentation to the transportation specialist, or designee, for further handling in accordance with EH-4527, *Receipt, Transport, and Movement of Radioactive Materials* (PRS-RAD-1105, *Receipt, Transport and Movement of Radioactive Materials*), and TR-9503, *Shipping Samples from a Bechtel Jacobs Company Site* (PRS-WSD-9503, *Offsite Sample Shipping*).

5. Nondisposable sampling equipment, tools, or supplies shall be decontaminated in accordance with BJC-ES-2702, *Decontamination of Sampling Equipment and Devices* (PRS-ENM-2702, *Decontamination of Sampling Equipment and Devices*). Prior to transporting contaminated equipment, tools or supplies, contact the Transportation Specialist.

6. Any IDW generated by the decontamination process will be collected, segregated into appropriate containers, labeled, and disposed of (or treated prior to disposal) in accordance with the requirements in the WP. Prior to transporting IDW, contact the Transportation Specialist.

4.0 RECORDS

The Field Logbook along with sample container COA or certificates of cleanliness, and COC Form(s) are project records and shall be maintained according to BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

5.0 SOURCE DOCUMENTS

U.S. Environmental Protection Agency, November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, Section 5*. Region 4, Environmental Compliance Branch, Athens, GA.

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 2

DEFINITIONS

Equipment Rinsate Blank: A sample of analyte-free or organic-free water poured over and/or through decontaminated sampling equipment. The purpose of the equipment rinse blank is to assess the adequacy of the decontamination process.

Field Blank: A blank solution of analyte-free or organic-free water that is prepared in the field and subjected to all aspects of sample collection, field-processing, preservation, transportation, and laboratory handling as an environmental sample. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

Field Duplicate Sample: Samples are one of two types: subsample (replicated) or co-located. Subsamples are similar to a split sample except the same laboratory analyzes both samples. These samples do not assess site heterogeneity, only specific sample point heterogeneity. The material may be homogenized (except volatiles) before being divided. Co-located samples are two or more separate portions collected from side by side locations at the same point in time and space so as to be considered identical. These samples are used to assess precision of the total method, including sampling, analysis, and site heterogeneity. These separate samples are said to represent the same population and are carried through all steps of the sampling and analytical procedures in an identical manner.

Trip Blank: A sample that is prepared prior to the collection of VOCs or tritium and is stored with the investigative samples throughout the sampling event. These samples are then packaged for shipment with the other samples and submitted for analysis. At no time after their preparation are trip blanks to be opened before reaching the laboratory. If samples are to be shipped, trip blanks are to be provided with each shipment that contains VOC or tritium samples, but not for each cooler.

Split samples: Two or more representative portions taken from a sample in the field or laboratory, analyzed by at least two different laboratories. Prior to splitting, a sample is mixed (except volatiles) to minimize sample heterogeneity. These are quality control samples used to assess precision, variability, and data comparability between different laboratories.

Sample Handler: The person (or designee) responsible for ensuring that sampling activities are performed in accordance with the approved work package and associated procedures, and approving (or coordinating the approval of) deviations there from. Specific position titles with these responsibilities may be designated as: project technical lead, sampling manager, lead engineer, sampling team leader, or task leader.

Attachment A
DEFINITIONS/ACRONYMS
Page 2 of 2

ACRONYMS

AHA - Activity Hazard Analyisis

COA - Certificate of Analysis

COC - Chain-of-Custody

IDW - Investigation-Derived Waste

PEMS - Project Environmental Measurements System

PPE - Personal Protective Equipment

PRS - Paducah Remediation Services, LLC

QC - Quality Control

RCT - Radiological Controls Technician

RWP - Radiological Work Permit

VOC - Volatile Organic Compound

WP - Work Package

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Environmental Monitoring	**PRS-ENM-2708**	**REV. NO. 0**
SUBJECT MATTER AREA: Sampling	**PREPARER: Tracy Kulik**	**Page 1 of 14**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** Tracey Brindley (Signature on File in DCC) 3/5/07	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **CHAIN-OF-CUSTODY FORMS, FIELD SAMPLE LOGS, SAMPLE LABELS, AND CUSTODY SEALS**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 3/12/07**	
USQD/UCD No: USQD-PH-SITE-0037/R1-UCD-PH-SITE-0075/T1	**REQUIRED REVIEW DATE: 3/12/10**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of PRS documentation. This document replaces BJC-ES-2708, Rev. 0, *Chain of Custody Protocol for Environmental Sampling*.	All

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1.0 PURPOSE

The purpose of this procedure is to describe the use of COC forms to track samples and ensure the integrity of those samples by documenting possession of transfers from the time of collection to acceptance by the designated laboratory. It includes requirements for the generation, use, and completion of COC forms, sample labels, and custody seals.

2.0 SCOPE

The requirements of this procedure apply to sampling performed for PRS and its subcontractors at the Department of Energy-owned Paducah Site.

3.0 PRECAUTIONS AND LIMITATIONS

To simplify the COC record and minimize the potential for error and other problems, as few people as possible shall have custody of the samples.

Entries on the COC form and sample labels shall be made using black indelible ink. Entry errors shall not be erased, altered, or otherwise rendered illegible. Void entry errors by drawing a single line through the entry, and initial and date the correction. Insertions after the initial entry should be initialed and dated. Do not use correction tape and white-out.

4.0 PROCEDURE

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in parentheses will become the reference procedures once these procedures are approved and implemented by PRS.

4.1 Pre-Performance Activities

4.1.1 Individuals who record information on COC forms, sample labels, and custody seals shall be knowledgeable of the latest version of this procedure before beginning any activities.

4.1.2 Gather needed supplies prior to the start of work. The items listed in this section may be used as a guide; however, additional items may be needed as determined by the Work Package.

- COC forms generated from Paducah PEMS database
- Sample labels generated from Paducah PEMS database
- Custody seals, as defined in section 4.7
- Extra blank COC forms, sample labels, and custody seals
- Black indelible ink pens
- Clear tape to cover sample labels, if they are not waterproof
- Logbook

4.2 Chain-of-Custody Form Generation

4.2.1 The Sample Handler is responsible for collecting, properly handling, and maintaining and documenting custody of the samples until they are formally transferred to another person or the laboratory.

4.2.2 A separate COC form shall be used for each laboratory that will perform sample analysis. COC forms should be pre-printed from the Paducah PEMS database if possible. In the event Paducah PEMS is not accessible and sampling is required, an equivalent COC shall be used. See Attachment B for an example of a blank COC.

4.2.3 The COC form(s) typically includes the following information:

- Project Identification Number
- Sample ID Number
- Sampling description and location
- SOW number
- Lab COC Number
- Laboratory Name
- Sample Types
- Container Sizes and Types
- Number of containers
- Sample Preservatives
- Matrix Codes
- Analysis requested
- Date and time of collection
- Sampler's initials

4.3 Chain-of-Custody Form Completion

4.3.1 All entries on the COC shall be made using black indelible ink.

4.3.2 Documentation of COC is accomplished by using a COC form. The COC follows the sample from the time of collection to receipt by the laboratory that will analyze the sample and sometimes until the sample is disposed (see Attachment C for an example COC form generated from Paducah PEMS).

NOTE: Field sample logs have been used in the past to document initial custody, but no longer will be used.

4.3.3 Record the following information at the time of the sampling event:

- Date and time sampled (military time)
- Sample handler's initials
- Volume of sample (if required for specific analysis and calculations)
- Any relevant comments

4.3.4 When an ad hoc sample is collected in the field, record all of the required information based on the list in section 4.2.3, to the extent known, on a blank COC form (see Attachment B) and in the Field Logbook in accordance with BJC-ES-2700, *Field Logbooks*, (PRS-ENM-2700, *Logbooks and Data Forms*).

4.3.5 When samples are not collected, draw a "Z" line through the uncollected samples shown on the prepopulated COC form. Initial and date the "Z" line and provide an explanation as to why the sample was not collected.

The explanation shall be more descriptive than "not collected" or "not needed" or "could not collect." Acceptable explanations must state why the sample was not collected, why the sample was not needed, or why the sample could not be collected. Examples of acceptable explanations include, but are not limited to these:

- Not collected due to poor recovery from the boring;
- Not needed because sample is a matrix spike; and
- Could not be collected because the well was dry.

4.4 Sample Label Generation

4.4.1 Sample labels are required to provide identification of samples collected for analysis at laboratories. When *in situ* measurements are made, the data are recorded directly in the logbook at the time of sampling along with the identifying information and any field observations.

4.4.2 Sample labels should be preprinted from the Paducah PEMS database if possible. In the event Paducah PEMS is not accessible and sampling is required, a Paducah PEMS equivalent label shall be used. See Attachment D for an example of a blank sample label. The following information typically will be included on the label:

- Project identification number
- Sample identification number
- Sample location ID
- Lab COC number
- Matrix Code
- Analysis to be performed
- Container size and type
- Preservatives used
- Laboratory name
- Date and time of collection
- Samplers initials
- NCS exempt designation of "PF" or "NCS Exempt" (contact the data manager for guidance if this information is unknown)

4.4.3 When an ad hoc sample is collected in the field, record all of the required information, to the extent known, based on the list in section 4.4.1 on a blank label (see Attachment D) and in the Field

Logbook (see BJC-ES-2700, *Field Logbooks*, [PRS-ENM-2700, *Logbooks and Data Forms*]).

4.5 Sample Label Completion

4.5.1 All entries on labels shall be made using black indelible ink.

4.5.2 Sample containers should be labeled prior to collection of the sample, to the extent feasible.

4.5.3 The following information shall be filled in at the time of sample collection:

- Name or initials of sample collector(s),
- Date and time (military time) of sample collection.

4.6 Special Sample Labels Required

If the samples to be collected contain material that exhibit any characteristic of hazardous waste, such as ignitability, corrosivity, reactivity, or toxicity, or is from a known or suspected asbestos or PCB source, contact the waste management group for guidance on any special labels that may be required before the samples are collected. Appropriate labels are applied based on process knowledge, source, or waste container labeling.

4.7 Custody Seals

4.7.1 In this document, custody seals include tape-like seals, tamper indicating tape, and tamper indicating devices that must be broken or removed to open the container after they are applied. Custody seals are used to guard against tampering and as a means to observe visually if tampering has occurred.

4.7.2 Any automatic composite sampler shall be secured with a custody seal or padlock to control access to the sample during collection.

4.7.3 **IF** samples are kept under positive control, as defined in Section 4.8, from the time of collection until the samples are delivered to the laboratory for analysis, **THEN** custody seals <u>are not</u> required.

4.7.4 **IF** the samples are going to be shipped or cannot be kept under positive control, as defined in Section 4.8, from the time of collection until the samples are delivered to the laboratory for analysis, **THEN** custody seals <u>are</u> required, and the following steps shall be followed.

1. As appropriate, use the following:
   - Custody seals with adhesive backs (See Attachment D for an example)
   - Tamper indicating tape
   - Tamper indicating devises such as a uniquely numbered

zip tie style (or similar) band, or
- Padlock

2. **IF** an adhesive backed custody seal is used, **THEN** sign and date the custody seal.
3. **IF** a zip tie style (or similar) band tamper indicating device is used, **THEN** record the unique identification number(s) on the COC and in the logbook.
4. Attach the seal or tamper indicating device to the container across the opening(s), so that the seal must be broken or removed to open the container.

4.7.5 **IF** a sample or shipping container must be opened as part of the sampling or shipping process (e.g., filtering a sample, adding additional ice to a composite sampler, adding additional materials to shipping container), **THEN** a new custody seal shall be applied. Record the action on the COC and in the logbook.

4.8 Positive Control

Maintain "positive control" of samples and COC forms from the time of collection until transfer to another custodian. "Positive control" requires one or more of the following:

- Physical possession;
- Visual control/oversight;
- Secured storage (i.e., lock and key) that only personnel authorized to handle the samples and COC forms can obtain keys to access; or
- Located in a secure area, with access to that area restricted to personnel authorized to handle the samples and COC forms.

4.9 Custody Transfer

4.9.1 To simplify the COC record, as few people as possible should handle the samples. Transfer of samples between field personnel in the same work group or between lab personnel in same work group does not need to be documented on the COC form; however, positive control of the samples must still be maintained. Before custody is transferred, ensure completeness of COC records.

4.9.2 When sample custody is transferred (e.g., for further processing, packaging, or transporting), the Sample Handler shall sign the form as "relinquished by" and enter the date and time. The receiving person shall verify sample container integrity and completeness of the COC form(s), sign the form as "received by" and enter the date and time of receipt. Each custodian must maintain positive control (see Section 4.8) of the samples and COC forms.

4.9.3 **IF** custody is transferred directly to another person, **THEN** the date/time will be the same for both the relinquished and received signatures.

4.9.4 **IF** the samples are shipped off-site, **THEN** the date/time will NOT be the same for the relinquished and received signatures.

4.9.5 On-site Laboratory Analysis

4.9.5.1 Transfer the samples and the original COC forms to the laboratory Sample Custodian.

4.9.5.2 Obtain a copy of the COC record and forward a copy of the signed COC record to the PRS Data Manager.

4.9.6 Off-site Laboratory Analysis

4.9.6.1 Common carriers (e.g., Federal Express), are not required to sign the COC record. When samples are shipped to an off-site for analysis, the COC record is signed by the laboratory Sample Custodian upon receipt at the laboratory; therefore, the date/time will not be the same for the relinquished and received signatures.

4.9.6.2 **IF** the samples require off-site shipment, **THEN** place the original COC form in a watertight bag and secure the bag inside the shipping container. Ensure custody seals are applied to the containers in accordance with Section 4.7.

4.9.6.3 Process the off-site shipment according to the applicable Department of Transportation regulations. Follow PRS procedures for shipment to the laboratory for analysis.

4.9.6.4 When the signed COC is received from the off-site laboratory, forward a copy of the signed COC record to the PRS Data Manager.

4.10 Post-Performance Activities

Submit a copy of the COC form to the PRS Data Manager for entry into Paducah PEMS.

5.0 RECORDS

Once the COC form is completed, it becomes a QA record and a copy of each form shall be maintained according to BJC-OS-1001, *Records Management, Including Document Control* (PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*).

6.0 SOURCE DOCUMENTS

U.S. Environmental Protection Agency, November 2001. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, Section 3.5. Region 4, Environmental Compliance Branch, Athens, GA.

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 1

DEFINITIONS

Ad Hoc Sample - Unplanned sample.

Chain-of-Custody - A process used to document the transfer of custody of samples from one individual to another from the time of collection until final disposition.

Custody - That process of assuring positive control (see Section 4.8 for more information) of a sample's integrity from the time of collection to receipt by the laboratory that will analyze the sample and sometimes until the sample is disposed. Documentation of COC is accomplished by using a COC form.

Custody Seals - A tape-like seal, tamper indicating tape, or tamper indicating device that must be broken or removed to open the container after it has been affixed. Custody seals are used to guard against tampering and as a means to observe visually if tampering has occurred.

***In Situ* Measurements** - Field measurements of sample characteristics taken and recorded at the time of sampling. Examples of *in situ* measurements include pH, temperature, dissolved oxygen, conductivity, and flow measurement.

Logbook - A bound book with sequentially numbered pages used to create a permanent, near real-time record of activities and conditions, significant events, observations, and measurements that occur during each day of field activities.

Project Manager - The person (or designee) responsible for ensuring that activities are performed in accordance with the current, approved plans or other governing documents and associated procedures. The project manager approves (or coordinates the approval of) deviations from the approved plans or other governing documents.

Sample Handler - The person who collects the sample and maintains custody of the sample following the requirements of this procedure until custody is transferred to another person. This person is responsible for overseeing the possession, exchange, and storage of the samples and COC forms.

ACRONYMS

BJC - Bechtel Jacobs Company LLC
COC - Chain-of-Custody
FSL - Field Sample Log
PEMS - Project Environmental Measurements System
PRS - Paducah Remediation Services, LLC
SOW - Statement of Work
NCS - Nuclear Criticality Safety
PF - potentially fissile

Attachment B
EXAMPLE OF A BLANK CHAIN-OF-CUSTODY FORM
Page 1 of 4

(Page 2 contains a full size copy of a blank COC form, which may be used directly. The COC form has no headers.)

Sample Chain-of-Custody Record

Sampling Description: _____

Sample ID: _____

Date/Time Sampled: _____

Project ID: _____

Sampler: _____

Station: _____ **Laboratory:** _____

LAB COC NO.: _____

Turnaround: _____

Sample Relinquished By _____ Date/Time _____

Received By _____ Date/Time _____

Sample Relinquished By _____ Date/Time _____

Received By _____ Date/Time _____

Sample Relinquished By _____ Date/Time _____

Received By _____ Date/Time _____

Sample Relinquished By _____ Date/Time _____

Received By _____ Date/Time _____

Analysis Requested _____

Matrix Code _____ Bottle Type _____ Bottle Size _____ No. of Bottles _____ SOW No. _____

Preservatives _____ Sample Type _____

Miscellaneous: _____

Analysis Requested _____

Matrix Code _____ Bottle Type _____ Bottle Size _____ No. of Bottles _____ SOW No. _____

Preservatives _____ Sample Type _____

Miscellaneous: _____

Analysis Requested _____

Matrix Code _____ Bottle Type _____ Bottle Size _____ No. of Bottles _____ SOW No. _____

Preservatives _____ Sample Type _____

Miscellaneous: _____

Attachment B
EXAMPLE OF A BLANK CHAIN-OF-CUSTODY FORM
Page 3 of 4

Directions for Completing a Blank Chain-of-Custody Form

Most of the information needed below can be obtained from the data manager or the laboratory coordinator if it is not already known.

Sampling Description – Describe what is being sampled (e.g., Asbestos sampling for doors in tank farm area).

Sample ID – Enter the unique sample ID number (e.g., DD06ASBDR-001D).

Date/Time Sampled – Enter the date and time that the sample was collected (e.g., 07/06/06 / 1245).

Project ID – Enter the Project ID for the sample (e.g., DD06-ASBDR).

Sampler – Enter the initials of the person who collected the sample.

Station – Enter the identifying location/station number (e.g., AHV14, MW389, WASTE, etc.).

Laboratory – Enter the Laboratory that will analyze the sample (e.g., PGDP, PORTS, PARAGON, etc)

Lab COC No. – Enter the Lab COC No. (e.g., DD06-ASBDR).

Turnaround – Enter the turn around time for the sample analysis (e.g., 14 days, 28 days, etc.).

Analysis Requested – Enter the analysis paragroup ID for the analysis that the lab will perform (e.g., DD-ASBESTOS, NEW-TC-99-PGDP, etc).

Matrix Code – Enter the matrix code for the material being sampled from the list below.

MATRIX CODE	DESCRIPTION
AIR	Air
FILTER	Filter
GAS	Identifiable non-air gas, or unidentifiable gas
LIQUID	Identifiable non-water liquid, or unidentifiable liquid
OIL	Oil
SD	Sediment
SLUDGE	Sludge
SOIL	Soil
SOLID	Identifiable non-soil solid, or unidentifiable solid
TISSUE	Tissue
WATER	Water (QC)
WG	Groundwater

Attachment B
EXAMPLE OF A BLANK CHAIN OF CUSTODY FORM
Page 4 of 4

MATRIX CODE	DESCRIPTION
WIPE	Wipe
WS	Surface Water
WW	Waste Water

Bottle Type – Enter the type of bottle that will be used for the sample (e.g., wide mouth glass).

Bottle Size – Enter the size of bottle that will be used for the sample (e.g., 2 ounce).

No. of Bottles – Enter the number of bottle that will be collected (e.g., 3).

SOW No. - Enter the SOW number for the sample (e.g., DD06-39).

Preservatives – Enter they type of preservative used for the sample (e.g., None, HCL pH<2, 4°C, etc).

Sample Type – Enter the sample type for the sample from the list below.

SAMPLE TYPE	DESCRIPTION
FB	Field Blank - a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected.
FR	Field Duplicate - two or more samples collected at the same sampling location either side by side or one immediately following the other.
FTB	Filter Blank - analyte-free water passed through a filter and collected in a sample container.
REG	Regular - primary sample collected for analysis.
RB	Refrigerator Blank - analyte-free water that is used to detect any cross contamination of samples stored in the laboratory refrigerator.
RI	QC Equipment Rinsate/Decontamination - a sample collected using analyte-free water which has been run over/through sample collection equipment used to determine if contaminants have been introduced by contact of the sample medium with sampling equipment.
TB	Trip Blank - a sample which is prepared prior to the sampling event and is stored with the investigative samples throughout the sampling event used to determine if samples were contaminated during storage and/or transportation to the laboratory.

Miscellaneous – Enter any other important information or comments regarding the sample.

Attachment C
EXAMPLE OF A CHAIN-OF-CUSTODY GENERATED FROM PEMS
Page 1 of 1

Sample Chain of Custody Record

Page ____ of _____

NW Tc-99/TCE - 30-Day TAT - EQ & Well Vaults

Sample ID HV082EW1-0806
Date/Time Sampled
Project ID NWOPS06-11 Sampler:_____
Station HV082
LAB COC NO.: NWOPS06-11 PGDP

Planned Sampling Date: 8/1/2006

Chain of Custody

Sample Relinquished By		Date/Time	
Received By		Date/Time	
Sample Relinquished By		Date/Time	
Received By		Date/Time	
Sample Relinquished By		Date/Time	
Received By		Date/Time	
COC Relinquished By		Date/Time	
Received By		Date/Time	

NEW-VOA-PGD Matrix: WG

Bottle: 40 ml VOA	Pres: HCL pH< 2, 4C	.2 ml	Volatiles Organics
Bottle: 40 ml VOA	Pres: HCL pH< 2, 4C	.2 ml	Volatiles Organics
Bottle: 40 ml VOA	Pres: HCL pH< 2, 4C	.2 ml	Volatiles Organics

SOW Numbers: NWOPS06-02 NA

8260	Trichloroethene

NEW-TC-99-PGD Matrix: WG

Bottle: 1000 ml plastic	Pres: HNO3 pH<2	2 ml

SOW Numbers: NWOPS06-02 NA

RL-7100	Technetium-99

Miscellaneous:_____

Attachment D
EXAMPLE OF A BLANK LABEL AND CUSTODY SEAL
Page 1 of 1

Blank Label – These labels may be printed onto sticker paper or shrunk and copied onto sticker paper for use as needed.

Project ID: _____
Sample ID: _____
Lab COC No.: _____
Matrix Code: _____ **Sampler:** _____
Location ID: _____ **Lab:** _____
Analysis Requested: _____
Date/Time Sampled: _____
Bottle Type: _____ **Bottle Size:** _____
Preserv. _____ **NCS Exempt** ☐ **PF**☐

Project ID: _____
Sample ID: _____
Lab COC No.: _____
Matrix Code: _____ **Sampler:** _____
Location ID: _____ **Lab:** _____
Analysis Requested: _____
Date/Time Sampled: _____
Bottle Type: _____ **Bottle Size:** _____
Preserv. _____ **NCS Exempt** ☐ **PF**☐

Project ID: _____
Sample ID: _____
Lab COC No.: _____
Matrix Code: _____ **Sampler:** _____
Location ID: _____ **Lab:** _____
Analysis Requested: _____
Date/Time Sampled: _____
Bottle Type: _____ **Bottle Size:** _____
Preserv. _____ **NCS Exempt** ☐ **PF**☐

Project ID: _____
Sample ID: _____
Lab COC No.: _____
Matrix Code: _____ **Sampler:** _____
Location ID: _____ **Lab:** _____
Analysis Requested: _____
Date/Time Sampled: _____
Bottle Type: _____ **Bottle Size:** _____
Preserv. _____ **NCS Exempt** ☐ **PF**☐

Custody Seal - These seals may be copied or printed onto sticker paper for use if needed. Other similar custody seals also are acceptable.

PADUCAH Remediation Services
A Portage Shaw Joint Venture Company

CUSTODY SEAL

Signature:_____ Date:_____

PADUCAH Remediation Services
A Portage Shaw Joint Venture Company

CUSTODY SEAL

Signature:_____ Date:_____

PADUCAH Remediation Services
A Portage Shaw Joint Venture Company

CUSTODY SEAL

Signature:_____ Date:_____

PADUCAH Remediation Services
A Portage Shaw Joint Venture Company

CUSTODY SEAL

Signature:_____ Date:_____

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED:

Paducah Site-Wide
Procedure Type
(Company, Functional, Project, Subcontractor)

PA-5107: Inorganic Data Verification & Validation
(Procedure Number and Title)

4 3/17/1999 PA-4306
(Procedure Date)

Rev. 0
(Rev. Number)

Reason for Changes: Transitioning BJC Procedures

SECTION	DESCRIPTION OF CHANGE
General	When reference is made to Lockheed Environmental Management and/or Bechtel Jacobs LLC personnel or contract, change the reference to Paducah Remediation Services (LLC) personnel or contract. Note that the procedure numbers will not change until after the first revision by PRS LLC.
General	Change Bechtel Jacobs LLC Environmental Management (EM) Projects to Paducah Remediation Services (LLC) Environmental Management (EM) Projects.
General	Attached is a cross walk from the Lockheed Environmental Management and the old Bechtel Jacobs LLC procedure numbers to the current Bechtel Jacobs LLC procedures. The procedure will be re-numbered with it is revised after transition.
Introduction	Delete the following sentence "This procedure applies to all PCDD/PCDF data verification and validation activities performed by or for the ER Program at the East Tennessee Technology Park (ETTP), Oak Ridge National Laboratory (ORNL), Y-12 Plant (Y-12), Paducah Gaseous Diffusion Plant (PGDP), and Portsmouth Gaseous Diffusion Plant (PORTS) sites, as well as those performed by or for ER's Off-Site Program." And replace it with the following "This procedure applies to all PCDD/PCDF data verification and validation activities performed by or for the ER Program at the Paducah Gaseous Diffusion Plant (PGDP) sites, as well as those performed by or for ER's Off-Site Program."

II Reference A. Source Documents	This procedure was last revised in 1999 and the source data lists Martin Marietta Energy Systems Inc. procedures It is anticipated that PRS LLC will review and up date this procedure, and at that time update the references to match the existing work flow.
II Reference B. Other Documents	This procedure was last revised in 1999 and some of the cited references have been revised and/or superseded. It is anticipated that PRS LLC will review and up date this procedure, and at that time update the references to match the existing work flow.

Initiator/Originator: Darryl Porter / [signature] — 4/7/06
(Print Name / Sign Name) — Date

REVIEWED BY: [signature] — 4/7/06
Discipline/Task Manager — Date

APPROVED BY: Diane McDaniel — 4/7/06
Project Manager — Date

BJC to PRS Position/Title/Organization Matrix
for PA-5107 Inorganic Data Verification and Validation

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
Data Manager	→	Data Manager/Network Specialist
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I. PURPOSE/SCOPE

This procedure defines the minimum requirements, responsibilities and methodology for the inorganic data verification and validation processes within the Bechtel Jacobs Company Environmental Restoration (ER) Program Organization. This procedure applies to all inorganic data verification and validation activities performed by or for the ER Program at the East Tennessee Technology Park (ETTP), Oak Ridge National Laboratory (ORNL), Y-12 Plant (Y-12), Paducah Gaseous Diffusion Plant (PGDP), and Portsmouth Gaseous Diffusion Plant (PORTS) sites, as well as those performed by or for ER's Off-Site Program.

This procedure provides requirements for developing and implementing a validation methodology for inorganic Contract Laboratory Program (CLP) and SW-846 (6010 and 7000 series) analytical methods primarily for analytes in aqueous and soil/sediment matrices. It is flexible enough to allow evaluation of data useability for project-specific Data Quality Objectives (DQO). Data produced by analytical methods for which this procedure provides limited guidance (i.e., Methods for Chemical Analysis of Water and Wastes, (EMSL-CI), EPA-600/4-79-020, Revised March 1983 and 1979 or "Superfund Low Concentration Statement of Work" methods) may necessitate development of modified criteria from this procedure; however, the general validation strategy outlined in this document should be applicable.

Specifications in this procedure should be incorporated into project documentation such as the Quality Assurance Project Plan, into contractual Statements of Work (SOW) between the project and the analytical laboratories, and into contractual validation SOWs between the project and the firm chosen to validate data. If validation is performed by individuals within the project, the SOW is not required but a mechanism to specify validation requirements is recommended. This procedure shall be used as a baseline to create project-specific checklists needed to perform inorganic data verification and validation. Guidance in developing and implementing a validation plan is provided in the procedure ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects". Examples of checklists for implementing this procedure are provided in Appendixes A and B; current versions of these checklists are readily available from the ER Data Quality Program Forms Reference Library (423/241-4677) in either hardcopy or electronic format.

In this procedure, the words "shall" and "must" are used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission, neither a requirement nor a recommendation. In conformance to this procedure, all verification and validation shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

II. REFERENCES

A. Source Documents

Data Management Plan for the Environmental Restoration Program, ES/ER/TM-88.

"Comprehensive Data Validation," ER/C-S2201, Rev. 0, Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee, October 29, 1993.

"Data Validation Plans for Environmental Restoration Projects," ERWM/ER-P2213, Bechtel Jacobs Company, LLC, Oak Ridge, Tennessee.

B. Other Documents

USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis. ILM04.0.

USEPA Test Methods for Evaluating Solid Waste. SW-846 third edition, November 1986. (Through Final Update III, December 1996).

USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review. EPA/540/R-94/013, February 1994.

Data Quality Objectives Process for Superfund. EPA/540/G-93/071, September 1993.

Guidance for the Data Quality Objectives Process. EPA QA/G-4. August, 1994

III. DEFINITIONS

Qualifier definitions and reason codes are listed in Attachment C.

Affected Sample Result: An affected sample result is a sample result considered to be affected significantly by a quality deficiency and is qualified accordingly through analytical data validation.

Analytical Data Validation: Analytical data validation is a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Analytical Data Verification: Analytical data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract which is performed by either the data generator or by an entity external to the data generator.

Batch: A batch is a group of samples prepared at the same time in the same location using the same method, not to exceed 20 samples of similar matrix.

Case: A finite, usually pre-determined number of samples collected over a given time period from a particular site. A case consists of one or more sample delivery groups (SDGs).

Chain of Custody (COC): The COC is the history of the transfer of samples from the time of sample acquisition through archival and disposal of samples.

Continuing Calibration Blank (CCB): The CCB is a deionized water sample analyzed immediately following the continuing calibration verification, designed to detect carryover.

Continuing Calibration Verification (CCV): The CCV is a standard solution analyzed at a specified frequency during an analytical run to assure continued validity of the calibration curve.

Contract Required Detection Limit (CRDL): The CRDL is the minimum level of detection acceptable under the current contract.

Correctable Problem: Correctable problems are deficiencies within data packages which may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation.

- correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory

- correctable problems revealed during validation are those deficiencies with analyses that can be solved by either a second preparation and/or analysis of a sample.

Graphite Furnace Atomic Absorption (GFAA) spectrometry: The GFAA is an atomic absorption spectrophotometer equipped with a graphite furnace that ashes and subsequently atomizes analytical samples to detect the extent of absorbance of a predetermined wavelength of electromagnetic radiation.

Inductively Coupled Plasma (ICP): The ICP is a technique for the simultaneous multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique." Source: USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, ILM04.0.

Initial Calibration Blank (ICB): The ICB is a deionized water sample analyzed immediately following the Initial Calibration Verification Sample at the beginning of an analytical run.

Interference Check Sample (ICS): The ICS is a solution of interfering and analyte elements of known concentrations, used to verify the validity of the laboratory's interelement and background correction factors for ICP only.

Initial Calibration Verification (ICV): The ICV is a standard analyzed immediately following instrument or method calibration as the first sample in an analytical run to confirm the validity of the calibration curve.

Instrument Detection Limit (IDL): The IDL is the detection limit of a particular analytical instrument determined by multiplying by three the standard deviation obtained for the analysis of a standard solution at a concentration of 3X to 5X the estimated IDL on three nonconsecutive days with seven consecutive measurements per day.

Laboratory Control Sample (LCS): The LCS is a control sample of known composition. Aqueous and solid LCS are analyzed using the same preparation, reagents, and method employed for field samples.

Laboratory Duplicate: The laboratory duplicate is a randomly chosen split of a field sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

Linear Range Analysis (LRA): The LRA denotes the concentration range over which the ICP analytical curve remains linear.

Matrix Spike: The matrix spike is a split of a field sample in which one half of the split is spiked with a known amount of analyte of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation

Method of Standard Additions (MSA): The MSA is the procedure of adding 3 increments of a standard solution to sample aliquots of the same size.

Non-Correctable Problem: Non-correctable problems are deficiencies within data packages which preclude the evaluation of data quality by predefined criteria. Non-correctable problems may be revealed during both data verification and data validation.

Quality-indicator Sample: Quality-indicator samples are those samples made ready in the laboratory which provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

Relative Percent Difference (RPD): RPD is the measure of precision between two values, defined as the absolute value of the difference between two values divided by the mean of the two values.

Relative Standard Deviation (RSD): RSD is the measure of precision between multiple values, defined as the standard deviation of multiple values divided by the mean of the values.

Required Detection Limit (RDL): The RDL is a contractually-specified detection limit which, under typical analytical circumstances, should be achievable.

Sample Delivery Group (SDG): An SDG is defined by one of the following, whichever occurs first:

- Case of field samples
- Each 20 field samples within a case
- Each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the SDG.

Sample Result: A sample result, as described in this procedure, is a numeric denotation of the concentration, amount or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

Standard Reference Material (SRM): A SRM is a material or substance of which one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Technical Holding Time: For inorganics, technical holding time is the length of time, expressed in calendar days, from sample collection to sample analysis.

Traceable Reference Material (TRM): A TRM is a NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).

Turn-around Time: Turn-around time is contractually-specified as the length of time which elapses between laboratory receipt of the field samples and subsequent data receipt by the client.

Validation Statement of Work: The validation SOW is a document prepared by the ER project to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

Well Characterized Reference Material (WCRM): The WCRM may be derived from a field sample which has been well characterized through multiple analyses, providing a high level of confidence of the concentration in the sample. The WCRM may be submitted to NIST for characterization and classification as a TRM.

IV. GENERAL INFORMATION

A. Data Verification

Data verification is defined as a systematic process, performed by either the data generator (on-site or fixed base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

If verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

Data verification, at the project level, may be conducted either by the data verifier or by the data validator, whichever expedites the review process. If verification is conducted independently of the validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and statement of work, in terms of the required deliverables. The second activity entails various checks of the quality of the data, the purpose of which is to alert the validator that a non-correctable problem has occurred and that data may need qualification. If verification is conducted by the validator, the first activity is conducted similarly, but the second activity may result in prompt qualification of data.

Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an after-the-fact identification of non-correctable deficiencies.

A verification report is written by the data verifier and takes, as input, the steps in this procedure that are listed as "Verification." The data verifier does not qualify data if verification criteria are not met but indicates in the verification report the circumstances surrounding correctable and non-correctable problems in the data package which will be transferred to the validator for possible qualification. The verification report must be in a standard format, and must remain associated with the analytical data package. A verification report which includes all verification checks in this procedure is provided in Attachment A.

B. Analytical Data Validation

Analytical data validation, including field and laboratory data review, is defined as a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data which may result in qualification of the data. Data validation is not performed by the analytical laboratory. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent, and if present, the level of uncertainty associated with the measurement, and occurs prior to drawing a conclusion from a body of data. Analytical data validation includes a technical review of a laboratory data package covering the evaluation of quality control samples, the identification and quantitation of analytes, and the effect of deficiencies in quality on analytical sample data.

A validation report that includes the results of validation activities must be completed by the validator and takes, as input, the verification report and the steps in this procedure that are listed as "Validation." A validation report must be completed for every SDG that passes through validation. To expedite writing the validation report, comments must include explanations for qualification only if the reason codes do not adequately describe justification for qualification. Comments on data qualification for which reason codes adequately describe qualification reason are not necessary. A validation report which includes all validation checks in this procedure is provided in Attachment B.

Where professional judgement is used, a second opinion on choice of qualification of data should be sought. Consensus on professional judgement should be documented in the validation report.

V. REQUIREMENTS

A. General Requirements

1. This procedure will become effective thirty (30) days following the approval date to allow for distribution and initiation of training.

2. The actions described in this document must serve as the baseline for incorporation into project verification/validation activities. Project-specific verification/validation procedures applying to analytes and analytical methods not covered in this document must be submitted to the ER Data Quality Program for review and approval prior to use. Verification and validation activities subcontracted outside Bechtel Jacobs Company must adopt requirements consistent with those provided in this procedure.

B. Analytical Statement of Work

Analytical SOW Requirements:

Implementation of this procedure is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory Statement of Work (SOW). Deliverable requirements specified in the analytical SOW must be consistent with the requirements of this procedure.

C. Validation Statement of Work Requirements

The validation SOW must be written consistent with the requirements and specifications of this procedure. The validation SOW is prepared by the ER project team and functions as the mechanism by which validation implementation requirements are communicated from the project team to the validation organization.

D. Validation Data Qualification Requirements

When qualification conditions are met, a qualifier and reason code(s) shall be physically and/or electronically associated with the affected result(s). If more than one reason code is applied, each should be separated by commas. The listing of available reason codes is presented in Attachment C. If data are not qualified, an equals sign ("=") is entered on the sample result. As directed in the validation SOW or as appropriate, reason codes may be applied without qualifiers.

E. Validation Strategy Worksheet Requirements

The worksheet presented in Attachment D must be completed by the project to identify the working inorganic data validation approach. For worksheet items where direct implementation of this procedure is not appropriate, an alternative approach must be provided and must be submitted to the ER-Data Quality Program (DQP) for approval. Establishment of the data validation strategy and plan will be described in ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects".

F. Verification and Validation Reporting Requirements

The verification report (Attachment A) and validation report (Attachment B) must be completed for every SDG that passes through validation. Both the verification and validation reports must be completed regardless of who performs the reviews.

VI. RESPONSIBILITIES

The responsibilities outlined in this section do not preclude an individual assuming multiple responsibilities.

A. ER-DQP Data Validation Coordinator

The ER-DQP Data Validation Coordinator is responsible for approval of alternative and non-routine verification and validation activities, and for providing training and technical support to ER projects in the areas of data verification and validation.

Responsibilities listed below denote activities at the project-level:

B. Project Manager

The Project Manager is responsible for oversight of the validation activities occurring within the project as a result of implementation of the requirements of this document.

C. Data Verifier

The Data Verifier is responsible for performing the verification actions listed in this procedure, including all items in Sect. VII listed as "Verification" and the writing of a verification report.

D. Data Validator

The Data Validator is responsible for performing the validation actions listed in this procedure, including all items in Sect. VII listed as "Validation" and the writing of the validation report.

E. Data Validation Coordinator

The Data Validation Coordinator is responsible for developing a data validation plan for the project, for incorporating the requirements of this procedure into the data validation plan for the project, for completing the validation implementation form (Attachment D), and for interfacing with the laboratory concerning data package deficiencies.

F. Analytical Services Coordinator

The Analytical Services Coordinator is responsible for interfacing with the data validation coordinator to ensure that analytical statements of work incorporate deliverables required by this procedure so that data packages from the laboratory will be appropriate for verification and validation.

G. Records Management Coordinator

The Records Management Coordinator is responsible for interfacing with the data validation coordinator in transferring data packages and verification/validation reports, and in providing a system of storage and control of these documents.

H. Data Management Coordinator

The Data Management Coordinator is responsible for interfacing with the data validation coordinator in transferring validated sample data and in entering validated data into the project database.

VII. ACTION STEPS

The steps in this section define the processes of inorganic data verification and validation.

Refer to Attachment C for qualifier and reason code descriptions. Refer to Attachment E for guidance on qualification due to multiple quality deficiencies. Refer to Attachment F for a listing of relevant equations to use with this procedure.

A. Overall Implementation of Validation

The following is a step-by-step approach to implement analytical data verification and validation activities for ER projects.

1. Validation Strategy and Plan Development

   Data Validation Coordinator and Project Team

   Develop validation strategy based on inputs identified through the DQO process according to ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects".

   Complete the Inorganic Data Validation Implementation Form (Attachment D).

   Write a validation Statement of Work to communicate verification and validation requirements to the organization performing that work for the ER project.

2. Analytical Statement of Work

   Analytical Services Coordinator

   Write the analytical Statement of Work which communicates laboratory requirements from the ER project to the analytical laboratory.

3. Implementing Verification and Validation

   Verifier and Validator

   Implement verification and validation by completing the verification and validation reports (Attachments A and B, respectively).

B. Custody of Samples and Sample Documentation

The chain of custody (COC) is relevant to the representativeness of the sample to its origin. It is also legal documentation of sample possession. The COC documents the personal accountability of the samples, and any break in that signature series could result in jeopardizing the legal admissibility of the sample data.

While verification/validation cannot assure a sample has truly been in custody from the field to the final result, an evaluation of field and laboratory COCs will provide the best indicator. A sample is in custody if any of the following conditions are met:

- it is within one's possession;
- it is within one's view, after being in one's possession;
- it was in one's possession and then was secured to prevent tampering;
- it is placed in a designated secure area.

1. Verification

   Verification includes CLP Form I header checks for accuracy from the COC. If header information is incorrect when compared to the COC, the verifier will mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, every attempt should be made to verify the true identity of each sample. Custody problems will affect the representativeness of the sample(s).

Data Verifier

Trace the chain of signatures of all samples in the reporting batch from field sampling through receipt at the laboratory. If there is a break in the signature chain, indicate this problem in the verification report.

Verify the headers of sample results summary reporting forms against COCs for accuracy. If header information is incorrect when compared to the COC, mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, make every attempt to verify the true identity of each sample. If the identity of the sample can not be verified, note the occurrence on the verification report.

2. Validation

Data Validator

Qualify sample data that are not traceable through COC signature records "R."

C. Holding Times, Turn-around Times, and Sample Preservation

Holding times have been established by EPA to provide a maximum period of time in which an environmental sample will remain representative of its sampling location. Holding time begins when the sample is collected in the field, rather than time of sample receipt or date of digestion/distillation, and ends when the analysis has been initiated. If the final reported data results from a dilution or reanalysis of the sample, this analysis must have been completed within the holding time.

1. Deliverables

- Field COCs
- Laboratory COCs
- Dates of collection, preparation and analysis for all samples
- CLP Form XIV or equivalent for SW-846 methods.

2. Criteria

All samples must be preserved and must be analyzed within the holding time.

Table 1 - Holding Time Criteria (Water Samples)

Parameter	Holding time	Preservation
AA and ICP Metals	180 days	pH < 2, with HNO_3
Mercury	28 days	pH < 2, with HNO_3
Cyanide	14 days	pH > 12 with NaOH, Cool to 4°C ± 2°C
All Soil/Sediment	180 days	Cool to 4°C ± 2°C

The holding time criteria for metals listed in SW846 Update III, chapter 3, Table 3-I shall be applied to soils. Water holding time criteria may be applied to soils for other inorganic analytes; however, if those criteria are applied, an explanation for using water criteria must be provided in the validation report.

3. Verification

Verify the presence of the pertinent COCs.

Turn-around time is evaluated as **strictly a contractual issue**. An agreement should be established between the client and the laboratory concerning turn-around times for reported data. If turn-around times are not satisfied, a contractual mechanism should provide for appropriate action.

Data Verifier

Verify the presence of the pertinent COCs. Verify that sample results have been reported within the contractually-specified turn-around time. If data are noncompliant, note this in the verification report.

4. Validation

Data Validator

Qualify results ? IDL "J" and < IDL "UJ" if samples have not been preserved and/or holding time has been exceeded. Qualify data < IDL "R" if the reviewer determines that effects of holding time exceedence on sample results were significant.

If preservation documentation cannot be verified on the COC, the sample(s) must be considered unpreserved; however, if sample data do not seem affected by lack of preservation, data may not require qualification; however, place reason code.

D. Calibration

Instrument Calibration is established to ensure that the instrument(s) is capable of producing acceptable qualitative and quantitative data. If calibration fails, the instrument should be recalibrated. Initial calibration verification demonstrates that the instrument is capable of acceptable performance at the beginning of a run, and continuing calibration verification demonstrates that the instrument remains in control.

1. Deliverables

- CLP Form IIA, IIB, and XIV, or equivalent for SW-846 methods, for each initial calibration.
- Raw data for each initial calibration (required for confirmation).

2. Frequency

Table 2 - Calibration Frequency

Parameter	ILM04.0	SW-846
Instrument Calibration	Daily or once every 24 hours, whichever is more frequent, and every time the instrument is set up.	Daily or once every 24 hours
ICV	Immediately after instrument calibration.	Immediately after instrument calibration.
CCV	Mid-level standard every 10 samples or 2 hours, whichever is more frequent, and at the end of the analytical run.	Mid-level standard or QC check standard every 10 samples (for CN, every sample batch) and at the end of the analytical run.

3. Performance Criteria

Table 3 - Calibration Performance Criteria

Parameter	ILM04.0	SW-846
Initial Calibration		
AA metals	Blank and at least 3 standards, one at CRDL; r must be ≥0.995.	Blank and 3 standards; r must be ≥ 0.995.
ICP metals	A blank and at least one standard.	Blank and at least one standard.
Hg	Blank and 5-8 standards. r must be ? 0.995.	Blank and 5 standards. r must be ? 0.995.
CN	Blank and at least 3 standards, one at CRDL and one (midrange) distilled. r must be ? 0.995 for photometric determinations.	Blank and 6 standards.
ICV and CCV		
AA metals	Independent standard at a concentration other than that used for instrument calibration at every wavelength used for analysis. CCV analyzed after every 10 injections or every 2 hours, whichever is more frequent.	Calibration check standard at or near the mid-range. CCV analyzed after every 10 injections.
ICP metals		Reanalyze the highest mixed instrument calibration standard as if it were a sample.
Hg		Not specified.
CN	The standard must be distilled	Not specified.
Percent Recovery (Contractual criteria)		

AA metals	ICV & CCV: 90% - 110%	ICV: 90% - 110% CCV: 80% - 120%
ICP metals	ICV & CCV: 90% - 110%	ICV: 90% - 110% CCV: 90% - 110%
Hg	ICV & CCV: 80% - 120%	ICV: 90% - 110% CCV: 80% - 120%
CN	ICV & CCV: 85% - 115%	ICV & CCV: 85% - 115%

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, or if the required standard concentrations are not used, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "C07" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "C06" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality. However, if the instrument was not calibrated each time the instrument was set up, qualify the data "R."

   b. If the minimum number of standards was not used, and a sample result occurs outside the calibrated range of the instrument, qualify "R".

   c. If the midrange CN standard was not distilled, qualify all associated results "J."

   d. If inappropriate standard concentrations were used, and data quality is affected, qualify results > IDL "J" and results < IDL as estimated "UJ."

   e. If correlation coefficient is <0.995, qualify results > IDL "J" and results < IDL as estimated "UJ."

   f. The following table provides qualification action based on initial and continuing verification percent recovery:

Table 4 - ICV and CCV Qualification

Parameter	Percent Recovery (Validation Acceptance Criteria)	Sample Qualification	
		Result ≥ IDL	Result < IDL
AA and ICP CN Hg	111% - 125% 116% - 130 % 121% - 135%	J	no qualification
AA and ICP CN Hg	75% - 89% 70% - 84% 65% - 79%	J	UJ
AA and ICP CN Hg	< 75% < 70% < 65%	R	R
AA and ICP CN Hg	> 125% > 130% > 135%	R	no qualification

E. Blanks

Blank analysis determines the existence and magnitude of contamination problems. The calibration blanks (ICB and CCB) are used to ensure a stable baseline absorbance (GFAA), or emission (ICP) before analysis of analytical samples. The Preparation Blank (PB) is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process.

1. Deliverables

- CLP Form III or equivalent for SW-846 methods
- Raw data (required for confirmation)

2. Frequency

The following table provides frequency requirements for blanks:

Table 5 - Blank Frequency

Parameter	ILM04.0	SW-846
ICB	Immediately after instrument calibration.	Immediately after instrument calibration.
CCB	Every 10 samples or 2 hours, whichever is more frequent, and immediately following CCV.	Only specified for ICP - every 10 samples and at end of analytical run.
PB	Digest and analyze one PB for each matrix in the batch and for every 20 samples or per batch, whichever is more frequent.	Every sample batch.

3. Performance Criteria

a. The absolute value of the analyte concentration in ICB, CCB, or PB must be < IDL for CLP methods. For SW-846 methods, the calibration blank must agree within 3 standard deviations of the mean blank value.

b. All blanks in a SDG must be evaluated against sample results. Only the blanks associated with the field/QC samples must be used in determining data acceptability. Sample results must not be corrected by subtracting any blank value.

4. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

Data Validator

a. Place reason code "B07" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "B05" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality. If blank preparation and/or analysis frequency requirements have not been satisfied, sample data should only be qualified only if the deviation indicates an adverse effect on data quality.

b. Ensure that sample data have not been corrected by blank subtraction.

c. Qualification is considered necessary when samples are analyzed between either one or two significantly contaminated calibration blanks (according to the table below). Qualification shall be placed on samples respective to the "bracketing" calibration blank showing the highest level of contamination.

d. All samples prepared together shall be evaluated against the preparation blank.

e. When evaluating blank contamination in soil samples (or other solids), assure that qualification is based on the evaluation of calibration blanks, preparation blanks and analytical samples, all with the same units.

f. Ensure that dilution factors have been considered when evaluating samples for blank contamination.

g. The following table provides qualification guidance based on calibration and preparation blank data:

Table 6 - Blank Qualification

Calibration and/or Preparation Blank	Sample Result	Sample Qualification
Positive Value	< IDL	no qualification
> IDL	> IDL and < 5 X Blank	U
> IDL	> IDL and > 5X Blank	no qualification
*|-result| >2X IDL	< IDL	R
*|-result| >2X IDL	≥ IDL	professional judgement

* Negative blank values may be indicative of an out-of-control laboratory event. Negative blank results may indicate false negative sample values and should be evaluated on a per-sample basis.

h. Raw Data Confirmation

Confirm the presence (not necessarily the concentration) of analytes reported as detected in the blank from raw data.

F. Interference Check Sample (ICS)

The ICS verifies the validity of the laboratory's interelement and background correction factors for ICP only.

1. Deliverables

   - CLP Form IV or equivalent for SW-846 methods
   - Raw data (required for confirmation)

2. Frequency

   The ICSA and ICSAB must be analyzed at the beginning and end of the analytical run or a minimum of twice per 8 hour working shift, whichever is more frequent.

3. Performance Criteria

   Percent recovery of the ICSAB solution must fall within ±20% difference of the true value.

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "W03" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "W04" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality. If frequency requirements have not been satisfied, sample data should only be qualified only if the deviation indicates an adverse effect on data quality.

   b. In general, the sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICSAB.

   c. Sample interferant results must be ≥ the levels of the ICS for qualification of affected sample results. If samples show concentrations of Al, Ca, Fe, and/or Mg equal to or greater than those in the ICSAB, the following table provides guidance on qualification of sample data:

Table 7 - ICS Qualification

ICSAB Percent Recovery	Affected Sample Result(s)	Sample Qualification
>120%	<IDL	no qualification
>120%	>IDL	J
50% - 79%	<IDL	UJ
50% - 79%	>IDL	J
<50%	< or > IDL	R

d. If Al, Ca, Fe and Mg are present in the analytical samples at concentrations greater than the level in the ICS, or other elements are present in the sample at >10 mg/L, the reviewer should investigate the possibility of other interference effects by using Table 8. The analyte concentrations presented in the table should be considered estimated values. Therefore, estimate the concentration produced by an interfering element. If the estimated concentration is > 2X CRDL and also > 10% of the reported concentration of effected element, qualify the affected result "J."

e. If results > IDL are observed for elements which are not present in the EPA provided ICS solution, the possibility of false positives exist. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferants and with analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results > IDL "J." The following table can aid in identifying effects of interfering analytes.

Table 8 - Interelement Correction Factors

Analyte	Wavelength nm	Interferant									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Boron	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenu	203.030	0.05	--	--	--	0.03	--	--	--	--	--
m	231.604	--	--	--	--	--	--	--	--	--	--
Nickel	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Selenium	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Silicon	588.995	--	--	--	--	--	--	--	--	0.08	--
Sodium	190.864	0.30	--	--	--	--	--	--	--	--	--
Thallium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Vanadium	213.856	--	--	--	0.14	--	--	--	0.29	--	--
Zinc											

f. If negative results are observed for elements that are not present in the EPA ICS solutions, the possibility of false negatives in the associated samples may exist. If the absolute value of the negative results is > IDL, evaluate the associated sample data; if samples show comparable or higher levels of interferants, qualify results for the affected analytes < IDL "UJ".

g. If the samples have been analyzed for the presence of thorium or uranium, a cross-reference should be made to these fractions in order to determine whether the presence of these compounds may have presented interferences with the metal analyses. In evaluating overlapping emission lines, sample data may be qualified "J" or "R" through assessment of emission interferences.

G. Laboratory Control Sample (LCS)

The LCS serves to monitor the overall performance of all steps in the analysis, including sample preparation and instrumental analysis.

1. Deliverables

- CLP Form VII or equivalent for SW-846 methods
- Raw data (required for confirmation)

2. Frequency

The following table provides LCS frequency requirements:

Table 9 - LCS Frequency

Parameter	ILM04.0	SW-846
AA, ICP*, CN and Hg methods	One for each SDG or batch of samples digested per matrix, whichever is more frequent.	One for each sample batch.

* SW-846 methods do not clearly specify the frequency of the lab control sample preparation and analysis. It is recommended that the LCS be prepared and analyzed at the same frequency and evaluated against the same recovery criteria as CLP ILM04.0.

3. Performance Criteria

a. For AA, ICP, CN, and Hg CLP methods, the aqueous LCS percent recovery criteria are 80-120% (except for Ag and Sb). Soil percent recovery is based on the matrix used in preparation of the LCS. For SW-846 methods, the LCS recovery is based on control charting. The LCS recovery must be within 3 standard deviations of the mean.

b. The LCS may be provided to the laboratory by EPA. If unavailable, an ICV solution may be used.

c. Aqueous LCS may be used by the laboratory if a soil LCS is not available. The validator should make a note if a water LCS was used with soil field samples. If an aqueous LCS used for soil samples is out of percent recovery criteria, careful inspection must be made to determine the effect(s) on sample data. In comparing an aqueous LCS to soil sample data, ensure that units are comparable.

d. A LCS is not required for mercury and cyanide analysis in aqueous matrices.

4. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated and to the validator in the verification report.

5. Validation

Data Validator

a. Place reason code "L05" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "L03" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality. If LCS preparation and analysis frequency requirements have not been satisfied, sample data should only be qualified "J" only if the deviation indicates an adverse effect on data quality.

b. The following tables provide guidance concerning qualification of water and soil sample data based on LCS percent recovery (no LCS criteria applies to Sb or Ag):

Table 10 - LCS Water Qualification

Percent Recovery	Sample Result	Sample Qualification
50-79	>IDL	J
50-79	<IDL	UJ
>120	>IDL	J
>120	< IDL	no qualification
<50	affected sample result	R
SW-846 Methods		
> 3σ of mean	<IDL	no qualification
> 3σ of mean	>IDL	J
< 3σ of mean	<IDL	R
< 3σ of mean	>IDL	J

Table 11 - LCS Soil Qualification

Percent Recovery	Sample Result	Sample Qualification
outside EPA control limits	> IDL	J
greater than EPA control limits	< IDL	no qualification
lower than EPA control limits	< IDL	UJ

H. Laboratory Duplicate Analysis

A laboratory duplicate sample is analyzed to reflect precision of laboratory methods for each matrix.

1. Deliverables

- CLP Form VI or equivalent for SW-846 methods
- Raw data (required for confirmation)

2. Frequency

The following table provides Laboratory Duplicate frequency requirements:

Table 12 - Duplicate Frequency

Parameter	ILM04.0	SW-846
AA, ICP, CN and CVAA methods	One per each batch of 20 samples, or per each SDG, of a similar matrix and concentration and evaluated with the same instrumental method.	One per each sample batch or once per 20 samples, whichever is more frequent.

3. Performance Criteria

a. Samples identified as field blanks must not be analyzed as laboratory duplicates.

b. Relative Percent Difference must be within ±20% for aqueous samples (±35% for soils) for sample values ≥ 5X CRDL/Reporting Limit; and ±CRDL (± 2XCRDL for soils) for sample values < 5X CRDL/Reporting Limit, including the case when only one of the sample values is < 5X CRDL/Reporting Limit.

4. Verification

Data Verifier

a. Verify that field blanks were not analyzed as laboratory duplicates.

b. Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

Data Validator

a. Place reason code "D04" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "D02" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

b. The following table provides qualification guidance concerning duplicate precision:

Table 13 Duplicate Qualification

Sample Value	Duplicate Relative Percent Difference	Sample Qualification for results > IDL
? 5X CRDL	exceeds ± 20 % (water) exceeds ± 35 % (soil)	J
< 5X CRDL*	exceeds ± CRDL (water) exceeds ± 2 X CRDL (soil)	J

* Including the case when only one of the sample/duplicates is < 5 X CRDL.

I. Pre-digestion Spike Recovery

The pre-digestion spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

1. Deliverables

- CLP Form V or equivalent for SW-846 methods
- Raw data (required for confirmation)

2. Frequency

One pre-digestion spike must be analyzed with each SDG, or batch of 20 field samples, for each matrix, concentration and instrumental method. SW-846 AA/GF methods do not clearly specify the acceptance criteria for the pre-digestion spike. Therefore, it is recommended that pre-digestion spike be evaluated against the same recovery criteria as CLP ILM04.0.

3. Performance Criteria

a. Samples identified as field blanks must not be used for predigestion spike analysis.

b. Percent Recovery must be within control limits of 75% - 125%; however, if sample concentration is ? 4X spike concentration, spike % recovery criteria are not applicable.

4. Verification

Data Verifier

a. Verify that field blanks were not used for pre-digestion spike analysis.

b. Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

Data Validator

a. Place reason code "M05" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "M04" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

b. The following table provides qualification guidance for pre-digestion spike recovery:

Table 14 - Pre-digestion Spike Qualification

Percent Recovery	Sample Result	Sample Qualification
> 125%	<IDL	no qualification
> 125% or < 75%	>IDL	J
30% - 74%	<IDL	UJ
< 30%	>IDL	J
< 30%	<IDL	R

c. If the same sample that was used for duplicate analysis is used for pre-digestion spike analysis, spike calculations must be performed using the results of the "original sample".

d. For CLP methods, if any pre-digestion spike (analyzed with methods other than furnace) fails the criteria, except for Ag and Hg, check to ensure that the sample was analyzed as a post-digestion spike. If the laboratory failed to do this, a comment is needed in the validation report.

J. Furnace Atomic Absorption QC

Due to the nature of the Furnace AA technique, procedures unique to Furnace AA are applied to maintain an acceptable level of Quality Control. Duplicate injections and furnace post-digestion spikes establish the precision and accuracy of the individual analytical determinations.

1. Deliverables

   - CLP Form VIII, XIV or equivalents for SW-846 methods
   - Raw data (required for confirmation)

2. Frequency

   Duplicate injections are required on every analysis and post-digestion spikes are required on all analytical samples, preparation blanks, and laboratory control samples. MSA is applied as needed.

3. Performance Criteria

   a. The furnace AA scheme must be followed. This flow chart is provided in Appendix H.

   b. For sample results > CRDL, duplicate injection precision must agree within 20% Relative Standard Deviation (RSD). If the criteria is not met, the sample must be rerun once; if the criteria is still not met, the results are reported as-is.

   c. Analytical spike (post-digestion spike) percent recovery must be within the control limits, 85% - 115%.

   d. MSA is performed when sample absorbance (or concentration) is equal to or greater than 50% of the analytical spike absorbance (or concentration), and the analytical spike recovery is less than 85% or greater than 115%.

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "F13" on the affected data if non-correctable deliverable deficiencies have occurred, qualify only if the deviation indicates an adverse effect on data quality.

   b. For analytical samples > CRDL, if duplicate injection precision is outside the control limit, ± 20% RSD, qualify the sample result "J."

   c. The following table provides qualification guidance based on analytical spike percent recovery:

Table 15 - Analytical Spike Qualification

Percent Recovery	Sample Result	Sample Qualification
< 40%	> IDL	J
≥ 10% but < 40%	< IDL	UJ
< 10%	< IDL	R
* < 85% or > 115%	> IDL	J
* < 85% or > 115%	< IDL	UJ

* Applies only if sample absorbance (or concentration) is < 50% of analytical spike absorbance (or concentration)

d. If MSA was required but not performed, qualify affected sample results "J."

e. If MSA was spiked at levels other than 50, 100, and 150% the sample concentration, qualify data "J" only if this deviation indicates an adverse effect on data quality.

f. If the MSA correlation coefficient is < 0.995, qualify sample results "J."

K. Serial Dilution Analysis

Serial Dilution Analysis determines whether significant physical or chemical interferences from the matrix are present and are affecting the analysis of samples. This dilution is prepared from a selected digested sample. Serial dilution is only applicable for AA (SW-846) and ICP (ILM04.0 and SW-846).

1. Deliverables

- CLP Form IX or equivalent for SW-846 methods
- Raw data (required for confirmation)

2. Frequency

The following table provides Serial Dilution frequency requirements:

Table 16 - Serial Dilution Frequency

Parameter	ILM04.0	SW-846
AA	N/A	1 for each sample batch per matrix.
ICP	1 for each group of samples of similar matrix and concentration level or for each SDG, whichever is more frequent.	1 for every new or unusual sample matrix.

3. Performance Criteria

   a. Field Blanks and Preparation Blanks must not be used for the serial dilution analysis.

   b. The following table provides Serial Dilution performance criteria:

Table 17 Serial Dilution Performance Criteria

Parameter	ILM04.0	SW-846
AA	N/A	Analyte concentrations must be at least 25X IDL. 5X dilutions must agree within 10% difference of original or a post-digestion spike is required.
ICP	If analyte concentration is > 50X IDL, the 5X dilution must agree within 10% difference of the original.	If analyte concentration is > 10X IDL, the 5X dilution must agree within 10% difference of the original.

4. Verification

   Data Verifier

   a. Verify that field blanks and preparation blanks were not used for the serial dilution analysis.

   b. Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "W10" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "W12" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

   b. If percent difference is >10%, qualify "J" if appropriate (refer to Table 17).

   c. If negative interference is found (i.e. results of diluted samples are higher than the originals), data may be qualified as appropriate.

L. Sample Result Confirmation

Raw data should be requested based on the level of review by the validator and based on records requirements of the project.

The accuracy of sample result calculation by the laboratory can be addressed by two different techniques. The application of each technique depends on the laboratory's ability to minimize transcription during reporting and how familiar the project is with the performance of the laboratory. If sample results are produced primarily through software processing and minimal transcription is performed in the laboratory, the data system(s) can be evaluated during an audit or surveillance by performing two different tests on the software (1) supply the data system a consistent set of input designed to provide a consistent set of output, and (2) supply the data system a set of nonconforming data to test the error detection routines. An additional evaluation of the laboratory's software configuration control and security is also necessary. Through this technique, a high level of confidence can be gained in the laboratory's reporting techniques.

Calculations and rounding rules for analyte quantitation can be found in Attachment F.

Data Validator

If the laboratory has a high rate of manual transcription in generation of sample results, the project team may choose to manually recalculate sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contacting the laboratory may be necessary to resolve the problem. Data may be qualified "R" as a last resort if no actions can reproduce reported values.

If results are to be manually recalculated from raw data, the following strategy is recommended.

1. Examine raw data for anomalies.

2. Verify from raw data, two detected and non-detected results for ICP, two detected and non-detected results for GFAA, and two detected and non-detected results for CVAA. And two detected and non-detected results for CN analyses for each SDG. For aqueous sample results, use the concentration reported in raw data; for soils, use equation G.7 to convert concentrations in per-volume in raw data to per-weight:

3. Discrepancies may be resolved with the laboratory or may be resolved through a decision made by the validator and agreed upon through peer review, depending on the severity of the discrepancy(s).

4. Confirm from raw digestion logs that initial sample volumes are equivalent to final digestate sample volumes for ICP and GFAA digestions. If volumes differ, confirm that sample results have been corrected for the difference in final vs. initial volumes.

5. Confirm that results fall within linear range of the ICP (LRA) and within calibration range for other non-ICP parameters.

6. All analyses must fall within the calibration range. If outside, confirm that dilution results are corrected for dilution factor(s).

7. Confirm that results are > 5X ICP-IDL if ICP analysis results are used for As, Pb, Se, or Tl. However, if trace ICP analysis was used for these metals, confirm that the CRDL requirements were satisfied.

VIII. REQUIRED RECORDS

A. General Records

Records arising from the actions in this document are as follows:

- The verification report.
- The validation report.
- Copies of qualified, or unqualified results reports.

These records become part of the analytical data package and must be controlled through a records management system.

B. Quality Assurance Records

This procedure is considered a Quality Assurance Record and, as such, a record copy will be provided to the appropriate document management center (DMC) for retention according to the minimum schedule identified by DMC personnel.

IX. ADMINISTRATION

The Project Manager is responsible for ensuring that the requirements in this document are incorporated into project verification/validation plans. The data validation coordinator is responsible for incorporating the requirements into project plans and for interfacing with the ER-Data Quality Program and with other members of the project in its interpretation.

X. APPROVAL

This procedure must be approved by the Bechtel Jacobs Company ER Program Organization Director (or designee). Signature approval establishes the requirements contained in this procedure as being mandatory for the ER Program Organization.

Approved by

Approval date

Approval signature on file. Contact J. L. Hoyer at 423/574-7800.

XI. ATTACHMENTS

Attachment A: Data Verification Report
Attachment B: Data Validation Report
Attachment C: Qualifiers and Reason Codes
Attachment D: Inorganic Data Validation Implementation Form
Attachment E: Qualification Tables for Multiple Quality Deficiencies
Attachment F: Calculations and Equations

Attachment A

Inorganic Data Verification Report

Sample Delivery Group #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW#: _____

This checklist is to be used for verification of inorganic data packages, consistent with the procedure ES-A-0812. No qualifiers are placed through the process of verification.

Verified by:_____ Date:_____

	Y	N
Custody of Samples		
1. Are samples traceable through inspection of signature records on field and laboratory chains of custody?	___	___
Action: For any samples not traceable upon inspection of chains of custody, indicate the identities below and where the break in traceability occurred.		
Calibration		
2. Are Forms IIA, XIV or equivalent for SW-846 methods included in the data package?	___	___
3. Have frequency requirements for the ICV and CCV been satisfied?	___	___
4. Have the contract-required standard concentrations been used (or those specified in SW-846)?	___	___
Blanks		
5. Are Forms III, XIV, or equivalent for SW-846 methods included in the data package?	___	___
6. Have frequency requirements for the PB, ICB, and CCB been satisfied?	___	___
Interference Check Sample		
7. Is Form IV or equivalent for SW-846 methods included in the data package?	___	___
8. Have frequency requirements for the ICS been satisfied?	___	___
Laboratory Control Sample		
9. Is Form VII or equivalent for SW-846 methods included in the data package?	___	___
10. Have frequency requirements for the LCS been satisfied?	___	___
Laboratory Duplicate		
11 Is Form VI or equivalent for SW-846 methods included in the data package?	___	___
12. Have frequency requirements for the lab duplicate been satisfied?	___	___
13. Field blanks have not been used for the laboratory duplicate.	___	___
Pre-digestion spike		
14. Is Form V or equivalent for SW-846 methods included in the data package?	___	___
15. Have frequency requirements for the pre-digestion spike been satisfied?	___	___
16. Field blanks have not been used for the pre-digestion spike.	___	___
Furnace Quality Control		
17. Is Form I, VIII, XIV or equivalent for SW-846 methods included in the data package (Form VIII may not be present if MSA was not performed)?	___	___
Serial Dilution Analysis		
18. Are Form IX or equivalent for SW-846 methods included in the data package?	___	___
19. Have frequency requirements for the serial dilution analysis been satisfied?	___	___
20. Field blanks have not been used for the serial dilution analysis.	___	___

Action: Indicate non-correctable contract compliance deviations below.

_____

_____

_____

_____

_____

_____

_____

ATTACHMENT B

Inorganic Data Validation Report

Sample Delivery Group #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW#: _____

This checklist is to be used for validation of inorganic data packages, consistent with the procedure ES-A-0812. This checklist should be used only if the Inorganic Verification Report has been completed.

Instructions: If criteria are not met in this checklist, follow qualification provided in the "Q" column accompanied with the corresponding reason code in the "C" column. If necessary, additional codes may be placed.

Validated by:_____ Date:_____

1.0 Custody of Samples

Validation Step
Action: For any samples not traceable upon inspection of verification report and chains of custody, indicate the identities below and where the break in traceability occurred. Qualify samples not traceable "R (E01)." Indicate which samples were affected below.
Qualified Sample _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

2.0 Holding Times and Sample Preservation

Validation Step	Y	N	Q		C
			<IDL	≥IDL	
1. Were all samples analyzed within the appropriate holding time and preserved appropriately?			UJ or R	J	H03 H04 or H05

Action: Indicate affected samples below.

Sample	Holding Time Exceedence (Days)	Improper Preservation (check)
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Comments:_____

3.0 Calibration

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. Was the instrument(s) calibrated at the appropriate frequency?				--	--	C06
2. Were the minimum number of standards used in calibrating the instrument(s)?				--	--	C24
3. Was the midrange CN standard distilled?				UJ	J	C26
4. If contract non-compliant standard concentrations were used to make the calibration curve, do the concentrations characterize the range of interest in concentration?				UJ	J	C18
5. Were correlation coefficients ≥0.995?				J	J	C25
6. Were all ICV recoveries within control limits?				UJ/R	J/R	C20 or C21
7. Were all CCV recoveries within control limits?				UJ/R	J/R	C22 or C23

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Rejection

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

4.0 Blanks

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. Were PB, ICB, and CCB prepared and/or analyzed at the appropriate frequency?				--	--	B05
2. Were sample results verified as uncorrected for blank concentrations?				n/a	J	n/a
3. Were all blanks evaluated for contamination?				U	--	B01
4. Was the PB the same matrix as the analytical samples?				--	--	B02
5. Were negative concentrations in blanks evaluated?				U	n/a	B04
6. Were blank contaminant presences confirmed from raw data?				**	**	Q12 or B09

Action: Indicate affected samples only if data are rejected or if negative blank concentrations were the cause of qualification.

Sample	Reason for Rejection

Comments:_____

* Qualify only if the deviation indicates an adverse effect on data quality.

** Use professional judgement

5.0 Interference Check Sample

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. If the relevant reporting form was not included, place the following reason code on the affected sample data.				n/a	n/a	W03
2. Was the ICS analyzed at the appropriate frequency?				---	---	W04
3. Were the ICSAB %Rs within acceptance criteria?				UJ/R	J/R	W01 or W02
4. Were samples evaluated when elements other than Al, Ca, Fe, and Mg were present at levels >10 mg/L?				UJ/R	J/R	W05
5. Were samples evaluated for results for elements not present in the ICS solution?				---	J	W06
6. Were negative results for elements not present in the ICS solution evaluated?				UJ	---	W06
7. If samples were analyzed for thorium or uranium, were those results evaluated for high concentrations?**				n/a	J/R	W05

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Rejection

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

** Use professional judgement in qualifying due to potential interferences from high Th or U concentrations.

6.0 Laboratory Control Sample

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. If the relevant reporting form was not included, place the following reason code on the affected sample data.				n/a	n/a	L05
2. Was the LCS prepared and analyzed at the appropriate frequency?*				---	---	L03
3. Was the LCS matrix the same as the analytical samples?				UJ	J	L04
4. Were percent recoveries within acceptance criteria?				UJ/R	J/R	L01 or L02

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:_____

_____

_____

_____

_____

_____

* Qualify only if the deviation indicates an adverse effect on data quality.

7.0. Laboratory Duplicate

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. If the relevant reporting form was not included, place the following reason code on the affected sample data.				n/a	n/a	D04
2. Was the laboratory duplicate prepared and analyzed at the appropriate frequency?*				---	---	D02
3. Were reported precision estimates within acceptance criteria?				n/a	J	D03

Action: Indicate affected samples only if data were rejected.

Sample	Reason for Qualification

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

8.0 Pre-digestion spike

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. If the relevant reporting form was not included, place the following reason code on the affected sample data.				n/a	n/a	M05
2. Was the pre-digestion spike prepared and analyzed at the appropriate frequency?				---	---	M04
3. Were percent recoveries within acceptance criteria?				UJ	J/R	M01 or M02

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:_____

9.0 Furnace Quality control

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. Were duplicate injection criteria satisfied?				---	J	F01
2. Were post-digestion spike recovery criteria satisfied?				UJ/R	J	F02, F03
3. Were analytical spike percent recovery criteria satisfied?				UJ/R	J	F08, F09, F10, or F11
4. When MSA was required, was it carried out?				---	J	F04
5. Were MSA spike levels at 50, 100, and 150% of sample concentration?*				---	J	F06
6. Was MSA correlation coefficient ≥0.995?				---	J	F05

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

10.0 Serial Dilution Analysis

Validation Step	Y	N	n/a	Q		C
				<IDL	≥IDL	
1. If the relevant reporting form was not included, place the following reason code on the affected sample data.				n/a	n/a	W11
2. Was the serial dilution analyzed at the appropriate frequency?*				---	---	W12
3. Was serial dilution percent difference criteria satisfied?				---	J	W09

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Comments:_____

** Qualify only if the deviation indicates an adverse effect on data quality.

11.0 Sample Result Verification

Validation Step
For the following evaluations, some qualification of sample data may be possible. For contractual noncompliance, a validation code is placed if the occurrence is non-correctable.
1. Recalculate 2 detected and 2 nondetected results for AA, ICP, CVAA, and CN from each SDG from raw data.
2. Did recalculation confirm reported results? If not, increase the frequency of recalculations until adequate confidence is gained in the reported results.
3. Were initial sample volumes equivalent to final digestate volumes for ICP and GFAA digestions, or were sample results corrected for differences in volumes?
4. Were ICP results within linear range of the ICP?
5. Were GFAA and CVAA results within calibration range?
6. Were results from diluted samples corrected for the dilution factor?
7. Were ICP results for As, Pb, Se, and Tl >5X the ICP IDL?
8. If trace ICP analysis was used for As, Pb, Se, or Tl, were the CRDL requirements satisfied?
Action: Indicate instances of manual calculations not confirming reported results.
Sample _____ Reason for Qualification _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ Comments: _____ _____ _____ _____ _____ _____ _____

ATTACHMENT C

Data Validation Qualifiers and Reason Codes

Inorganic Analytical Data

Reason codes must be used with all qualifiers placed on analytical data.

U Analyte compound or nuclide considered not detected above the reported detection limit

J Analyte compound or nuclide identified; the associated numerical value is approximated

UJ Analyte, compound or nuclide not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.

R Result is not usable for its intended purpose

= "Equals" sign, indicates that no qualifier is necessary

DATA VALIDATION QUALIFICATION CODES

Inorganic Analytical Data

Blanks

B01 Concentration of contaminant in the blank at a level ? the qualification level.
B02 Blank was not the same matrix as the analytical samples.
B03 Gross contamination exists.
B04 Blank had a negative value > 2 X the IDL.
B05 Blanks were not analyzed at appropriate frequency.
B07 Blank data not reported.
B09 Other (describe in comments)

Calibration

C03 Initial calibration sequence was not followed as appropriate.
C06 Calibration or performance check was not performed at the appropriate frequency.
C07 Calibration data not reported
C08 Calibration not performed.
C10 Calibration standard matrix not the same as sample matrix
C18 Inappropriate standard concentrations
C19 ICV or CCV were not performed for analyte.
C20 ICV recovery was above the upper control limit.
C21 ICV recovery was below the lower control limit.
C22 CCV recovery was above the upper control limit.
C23 CCV recovery was below the lower control limit.
C24 Standard curve was established with fewer than the appropriate number of standards
C25 Calibration correlation coefficient was <0.995.
C26 Mid range cyanide standard was not distilled.
C31 Other (describe in comments)

Laboratory Duplicate

D02 Laboratory duplicate was not analyzed at the appropriate frequency
D03 Laboratory duplicate exceeds RPD criteria.
D04 Laboratory duplicate data not reported
D05 Other (describe in comments)

Evidentiary Concerns

E01 Custody of Sample in Question.
E02 Standard not Traceable.
E03 Other (describe in comments)

Furnace QC

F01 Duplicate injections were outside the control limit.
F02 Post digestion spike recovery was above upper control limit
F03 Post digestion spike recovery was below lower control limit
F04 MSA was required but not performed.
F05 MSA correlation coefficient was <0.995.
F06 MSA spikes were not at the appropriate concentration.
F07 Duplicate injection not performed.
F08 Furnace analytical spike percent recovery < 85%.
F09 Furnace analytical spike percent recovery > 115%.
F10 Furnace analytical spike percent recovery < 40%.
F11 Furnace analytical spike percent recovery < 10%.
F12 Furnace analytical spike not performed.
F13 Furnace QC data not reported
F14 Other (describe in comments)

General

G01 Professional judgement was used to qualify the data.
G02 Other (describe in comments)

Holding Times/Preservation

H03 Analysis holding times were exceeded.
H04 Analysis holding times were grossly exceeded.
H05 Samples were not preserved properly.
H06 Sample preservation can not be confirmed
H07 Sample temperature exceeded criteria prior to preparation
H08 Other (describe in comments)

Laboratory Control Sample

L01 LCS recovery above upper control limit.
L02 LCS recovery below lower control limit.
L03 LCS was not analyzed at appropriate frequency.
L04 LCS not the same matrix as the analytical samples.
L05 LCS data not reported
L06 Other (describe in comments)

Matrix Spike and MS/MSD

M01 MS and/or MSD recovery above upper control limit.
M02 MS and/or MSD recovery below lower control limit.
M03 MS/MSD pair exceeds the RPD limit
M04 MS and/or MS/MSD not analyzed at the appropriate frequency
M05 MS and/or MS/MSD data not reported
M06 Other (describe in comments)

Quantitation

Q04 Cross contamination occurred.
Q12 No raw data were provided to confirm quantitation.
Q14 Inappropriate aliquot sizes were used.
Q17 Negative result.
Q24 Raw data anomaly
Q25 Other (describe in comments)

ICP Requirements

W01 ICS recovery was above upper control limit
W02 ICS recovery was below lower control limit
W03 ICS data not reported.
W04 ICS not analyzed at the appropriate frequency.
W05 Interferance caused by metal other than ICSAB metal
W06 Negative value of interference check sample interferant.
W07 Post digestion spike recovery was above upper control limit
W08 Post digestion spike recovery was below lower control limit
W09 Serial dilution criteria were not satisfied.
W10 Serial dilution not performed.
W11 Serial dilution data not reported.
W12 Serial dilution not analyzed at the appropriate frequency.
W13 Other (describe in comments)

ATTACHMENT D

Inorganic Data Validation Implementation Form

Purpose: This worksheet is the mechanism to be used to identify and document the implementation of procedure ES-A-0812.

Instructions: One worksheet must be completed for each fraction and analytical method (e.g. metals by GFAA, metals by ICP). The completed worksheet shall be forwarded to the ER Data Quality Program for approval.

If the requirements of ES-A-0812 are adopted as-is, without the necessity of approved alternatives, enter "as-is" for review item "A" in the implementation column and complete the project sign-off section at the bottom of the form. No other entries are needed.

If project-specific implementations are necessary,

1) identify review items that will be directly implemented and enter "as-is" in the respective rows of the Implementation column.
2) identify review items that will not be considered by verifiers or validators during the implementation of this procedure, and enter "n/a" in the respective rows of the Implementation column and reference attached materials documenting the reason for the exclusion of these review items in the Comment column.
3) Identify review items that will be implemented with project-specified alternative actions and enter "Alternative" in the respective rows of the Implementation column. In the Comment column, reference attached materials that document the need for the alternative actions and specify the actions to be implemented upon approval. This alternative description should cover the following: Deliverables, Frequency, Performance Criteria, Verification step(s), Validation step(s).
4) Complete the project sign-off section at the bottom of the worksheet and forward to the ER Data Quality Program for approval.

Fraction and Method:_____

	Implement	Comment
1. All requirements of ES-A-0812 will be implemented	____	_____
2. Technical Holding Time	____	_____
3. Calibration	____	_____
4. Blanks	____	_____
5. Interference Check Sample	____	_____
6. Laboratory Control Sample	____	_____
7. Laboratory Duplicate	____	_____
8. Pre-digestion Spike	____	_____
9. Furnace Atomic Absorption QC	____	_____
10. Serial Dilution Analysis	____	_____
11. Sample Result Verification	____	_____
12. Manual Calculation of Results	____	_____

The _____ project will approach inorganic data validation consistent with ES-A-0812, and/or with specific alternative(s) described on the attached pages.

_____ _____

Signature Date

ATTACHMENT E

Guidance for Data Qualification

Due to Multiple Quality Deficiencies

This appendix provides guidance in the qualification of data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on potential effect on sample data. The effect of quality deficiencies may be applicable to only a single sample or to all samples within the reporting batch. A validation qualifier and associated reason code(s) should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

The following is a listing of data quality indicators and the probable effects on sample data.

Data Quality Indicator	**Effect on Sample Data**
Standard curve correlation coefficient	quantitative uncertainty
Initial or continuing calibration verification	quantitation
Method blank	positive bias
Interference check sample	positive or negative bias
Laboratory control sample	positive or negative bias
Laboratory duplicate	imprecision
Matrix spike (pre-digest spike)	positive or negative bias
Furnace QC	
MSA correlation coefficient	quantitative uncertainty
post-disgestion spike	positive or negative bias
duplicate injection	imprecision
Serial dilution	imprecision

In the instance of multiple quality deficiencies the validation qualifier and reason code(s) should be placed consistent with the acceptable level of uncertainty associated with the intended use of the data. The validation SOW should provide a summary of the intended use(s) of the data. (e.g., risk assessment, fate and transport modeling, waste management) to facilitate appropriate placement of validation qualifiers.

ATTACHMENT F

Rounding Rules

When using the calculations in this appendix, use the following rounding rules:

1. In a series of calculation, carry the extra digits through to the final result, then round off.
2. If the digit to be removed is less than 5, the preceding digit stays the same.

   If the digit to be removed is equal to or greater than 5, the preceding digit is increased by 1.

F.1 Initial/Continuing Calibration Verification Percent Recover

$$\%R = \frac{ICV_{Found}}{ICV_{True}} \times 100$$

Where,

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

True = concentration (in ug/L) of each analyte in the ICV or CCV source

F.2 Interference Check Sample Percent Recovery

$$\%R_{ICSAB} = \frac{AB_{Found}}{AB_{True}} \times 100$$

Where,

Found = concentration (in ug/L) of each analyte measured in the analysis of solution AB

True = concentration (in ug/L) of each analyte in solution AB

F.3 Laboratory Control Sample Percent Recovery

$$\%R_{LCS} = \frac{LCS_{Found}}{LCS_{True}} \times 100$$

Where,

Found = concentration (in ug/L for aqueous; mg/kg for solid) of each analyte measured in the analysis of LCS solution

True = concentration (in ug/L aqueous; mg/kg solid) of each analyte in the LCS source.

F.4 Laboratory Duplicate Relative Percent Difference

$$RPD = \frac{|R1 - R2|}{\bar{\times}_{R1,R2}} \times 100$$

Where,

R1 = first sample value (original)

R2 = second sample value (duplicate)

F.5 Pre-digestion Spike Percent Recovery

$$\%R_{PdS} = \frac{\text{Spiked Sample Result - Sample Result}}{\text{Spike Added}} \times 100$$

F.6 Serial Dilution Percent Difference

$$\%D = \frac{\text{Initial Result - Dilution Result}}{\text{Initial Result}} \times 100$$

F.7 Conversion of ug/L to mg/Kg

$$\frac{mg}{kg} = \frac{ug}{L} \times \frac{vol(mL)}{wt(g)} \times \frac{1L}{1000mL} \times \frac{1000g}{Kg} \times \frac{1mg}{1000ug}$$

ug/L = concentration from raw data

vol = digestate volume in Liters

wt = sample weight (1 g)

F.8 Conversion of soil/sediment wet weight to dry weight

$$\frac{mg}{kg} = \frac{mg}{kg}(wet) \times \frac{100}{\%solids}$$

%M = %moisture

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED:

Paducah Site-Wide
Procedure Type
(Company, Functional, Project, Subcontractor)

PA-5102: Radiochemical Data Verification and Validation
(Procedure Number and Title)

4-13-06
~~1/11/1999~~ 3-3-1999
(Procedure Date)

Rev. 0
(Rev. Number)

Reason for Changes: Transitioning BJC Procedures

SECTION	DESCRIPTION OF CHANGE
General	When reference is made to Lockheed Environmental Management and/or Bechtel Jacobs LLC personnel or contract, change the reference to Paducah Remediation Services (LLC) personnel or contract. Note that the procedure numbers will not change until after the first revision by PRS LLC.
General	Change Bechtel Jacobs LLC Environmental Management (EM) Projects to Paducah Remediation Services (LLC) Environmental Management (EM) Projects.
General	Attached is a cross walk from the Lockheed Environmental Management and the old Bechtel Jacobs LLC procedure numbers to the current Bechtel Jacobs LLC procedures.
Scope	Delete the following sentence "This procedure applies to all radiochemical data verification and validation activities performed by or for this Program at the East Tennessee Technology Park, Oak Ridge National Laboratory, Y-12 Plant, Paducah Gaseous Diffusion Plant, and Portsmouth Gaseous Diffusion Plant sites." And replace it with the following "This procedure applies to all radiochemical data verification and validation activities performed by or for this Program at the Paducah Gaseous Diffusion Plant sites."
References	This procedure was last revised in 1999 and some of the cited references have been revised and/or superseded. It is anticipated that PRS LLC will review and up date this procedure, and at that time update the references to match the existing work flow.

Pg 2

Initiator/Originator:	Darryl Poteet / [signature]	4/7/06
	(Print Name / Sign Name)	Date
REVIEWED BY:	[signature]	4/7/06
	Discipline/Task Manager	Date
APPROVED BY:	Diane M. Daniel	4/7/06
	Project Manager	Date

BJC to PRS Position/Title/Organization Matrix
for PA-5102 Radiochemical Data Verification and Validation

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
Data Validation Coordinator	→	Statistician/Validator
BJC Watershed Representative	→	Sr. Scientist
Data Validator	→	Statistician/Validator
Data Verifier	→	Data Entry Specialist
	→	
	→	
	→	
	→	
	→	
	→	
	→	
	→	
	→	
	→	
	→	
	→	
	→	

BECHTEL JACOBS
Bechtel Jacobs Company LLC

BJC-ES-01 Environmental Services Procedures Manual	ES-A-2209	REV. NO. 0
TITLE: RADIOCHEMICAL DATA VERIFICATION AND VALIDATION	EFFECTIVE DATE: 03/03/99	Page 1 of 73
	PREPARER/POC: Beth Mitchell	
	APPROVED BY/DATE: R.B. Barber 01/11/99 (Signature on File)	

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REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Replaces ERWM/ER-P2209	All

PURPOSE

This procedure defines the minimum requirements, responsibilities and methodology for radiochemical data verification and validation processes within the Bechtel Jacobs Company LLC (BJC-LLC) Environmental Management and Enrichment Facilities (EMEF) Program.

This procedure is applicable to radionuclide contaminants routinely analyzed by common radioanalytical methods primarily in aqueous and soil/sediment matrices. This procedure is applicable to environmental radiochemical data produced through methods which use instrumentation for detecting activity. This procedure is not applicable to mass spectrometric or fluorimetric methodologies.

Specifications in this procedure should be incorporated into project documentation such as the Quality Assurance Project Plan, into contractual Statements of Work (SOW) between the project and the analytical laboratories, and into contractual validation SOWs between the project and the organization chosen to validate data. If validation is performed by individuals within the project, the SOW is not required but a mechanism to specify validation requirements is recommended. This procedure shall be used as a baseline to create project-specific checklists needed to perform radiochemical data verification and validation. Guidance in developing and implementing a validation plan is provided in the procedure "Data Validation Plans for Environmental Restoration Projects," EMEF/P2213. Examples of checklists for implementing this procedure are provided in Appendixes B and C.

In this procedure, the words "shall" and "must" are used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission (neither a requirement nor a recommendation). In conformance to this procedure, all steps shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

SCOPE

This procedure applies to all radiochemical data verification and validation activities performed by or for this Program at the East Tennessee Technology Park, Oak Ridge National Laboratory, Y-12 Plant, Paducah Gaseous Diffusion Plant, and Portsmouth Gaseous Diffusion Plant sites.

OTHER DOCUMENTS NEEDED

Current version of "Terms and Conditions of the Analytical Support Agreement." Oak Ridge Sample Management Office

See Appendix A for a list of source documents and other references.

GENERAL INFORMATION

A. Data Verification

1. Data verification is defined as a systematic process, performed by either the data generator (on-site or fixed base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

2. If verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

3. Data verification, at the project level, may be conducted either by the data verifier or by the data validator, whichever expedites the review process. If verification is conducted independently of the validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and statement of work, in terms of the required deliverables. The second activity entails various checks of the data, the purpose of which is to alert the validator that a non-correctable problem has occurred and that data may need qualification. If verification is conducted by the validator, the first activity is conducted similarly, but the second activity may result in prompt qualification of data.

4. Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an after-the-fact identification of non-correctable deficiencies.

Verification Report

5. A verification report is written by the data verifier and takes, as input, the steps in this procedure that are listed as "Verification." The data verifier does not qualify data if verification criteria are not met but indicates in the verification report the circumstances surrounding correctable and non-correctable problems in the data package which will be transferred to the validator for possible qualification. The verification report must be in a standard format, and must remain associated with the analytical data package.

B. Analytical Data Validation

1. Analytical data validation including laboratory data review, is defined as a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data which may result in qualification of the data. Data validation is not performed by the analytical laboratory. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent, and if present, the level of uncertainty associated with the measurement, and occurs prior to drawing a conclusion from a body of data. Analytical data validation for radiochemistry includes a technical review of a laboratory data package covering the evaluation of quality control samples, the identification and quantitation of analytes, and the effect of deficiencies in quality control on analytical sample data.

Validation Report

2. A validation report that includes the results of validation activities must be completed by the validator and takes, as input, the verification report and the steps in this procedure that are listed as "Validation." To expedite writing the validation report, comments must include explanations for qualification only if the reason codes do not adequately describe justification for qualification. Comments on data qualification for which reason codes adequately describe qualification reason are not necessary.

3. Where professional judgement is used, a second opinion on choice of qualification of data should be sought. Consensus on professional judgement should be documented in the validation report.

REQUIREMENTS

A. General Requirements

1. The actions described in this document must serve as the baseline for incorporation into project verification/validation activities. Project-specific procedures applying to analytical methods not covered in this document must be submitted to the BJC-LLC Data Quality Program for review and approval prior to use. Verification and validation activities subcontracted outside Energy Systems must adopt requirements consistent with those provided in this procedure.

B. Radioanalytical Statement of Work

Radioanalytical SOW Requirements

1. Implementation of this procedure is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory Statement of Work (SOW). Deliverable requirements specified in the radioanalytical SOW must be consistent with the requirements of this procedure, and with the Analytical Master Specifications of the BJC Terms and Conditions.

Reporting Requirements

2. To enable performance of the evaluations in this procedure, all samples must be reported with a background-subtracted sample result, a ±2s counting uncertainty, and a ±2s Total Propagated Uncertainties (TPU) (factors for which are presented in Attachment G) and a Minimum Detectable Activity (MDA).

C. Validation Statement of Work Requirements

The validation SOW must be written consistent with the requirements and specifications of this procedure. The validation SOW is prepared by the ER project and functions as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

D. Validation Data Qualification Requirements

When qualification conditions are met, a qualifier and reason code(s) shall be physically and/or electronically associated with the affected result(s). If more than one reason code is applied, each should be separated by commas. The listing of available reason codes is presented in Attachment D. If data are not qualified, an equals sign ("=") is entered on the sample result. As directed in the validation SOW or as appropriate, reason codes may be applied without qualifiers.

E. Validation Strategy Worksheet Requirements

The worksheet presented in Attachment E must be completed by the project to identify the working radiochemical data validation approach. For items where direct implementation of this procedure is not appropriate, an alternative approach must be provided and must be submitted to the ER-DQP for approval. Establishment of the data validation plan is described in "Data Validation Plans for Environmental Restoration Projects," EMEF/P2213.

F. Validation Reporting Requirements

A verification report (Attachment B or equivalent) and validation report (Attachment C or equivalent) must be completed for every reporting batch that passes through validation. Both the verification and validation reports must be completed regardless of who performs the reviews.

WHAT TO DO

The Data Verifier performs all steps designated as "Data Verification" and the Data Validator performs all steps designated as "Data Validation."

Refer to Attachment D for qualifier and reason code descriptions. Refer to Attachment H for guidance on qualification due to multiple quality deficiencies.

Performer

1. Overall Implementation of Validation

The following is a step-by-step approach to implement analytical data verification and validation activities

Data Validation Coordinator and Project Team

a. Validation Plan Development

- Develop validation plan based on inputs identified through the DQO process according to "Data Validation Plans for ER Projects" EMEF/P2213.
- Complete the validation strategy worksheet (Attachment E).
- Write a validation Statement of Work to communicate verification and validation requirements to the organization performing that work for the ER project.

BJC Watershed Representative

b. Radioanalytical Statement of Work

- Write the radioanalytical Statement of Work which communicates laboratory requirements from the ER project to the radioanalytical laboratory.

Verifier and Validator

c. Implementing Verification and Validation

- Implement verification and validation by completing the forms (Attachments B and C, respectively).

Performer

2. Custody of Samples and Sample Documentation

The chain of custody (COC) is relevant to the representativeness of the sample to its origin. It is also legal documentation of sample possession. The COC documents the series of "ownership" of the samples, and any break in that signature series could result in jeopardizing the legal admissibility of the sample data.

While verification/validation cannot assure a sample has truly been in custody from the field to the final result, an evaluation of field and laboratory COCs will provide the best indicator. A sample is defined as being in custody if any of the following conditions are met:

- it is within one's possession;
- it is within one's view, after being in one's possession;
- it was in one's possession and then was secured to prevent tampering;
- it is placed in a designated secure area.

d. Verification

Verification of sample documentation includes result report header checks for accuracy from the COC. If header information is incorrect when compared to the COC, the verifier shall mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, every attempt should be made to verify the true identity of each sample, else sample representativeness is in doubt.

Data Verifier

Trace the chain of signatures of all samples in the reporting batch from field sampling through receipt at the laboratory. If there is a break in the signature chain, indicate this problem in the verification report.

Verify the headers of sample results summary reporting forms against COCs for accuracy. If header information is incorrect when compared to the COC, mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, make every attempt to verify the true identity of each sample; however, if sample representativeness remains in doubt, note the occurrence on the verification report.

e. Validation

Data Validator

Qualify sample data which is not traceable through signature records on COCs "R."

3. Holding Time and Turn-around Time

Holding time is defined for radiochemical data verification and validation as the period of time between sample collection and sample activity determination. Samples must be analyzed within a holding time precluding significant decay of short-lived target radionuclides. Holding time should be contractually specified to the laboratory. If holding times are not contractually satisfied, and if other technical corrective actions, such as increased count times or increased sample aliquot volumes or weights, have not been performed to compensate for long holding times, the required detection limit (RDL) may be exceeded.

However, as 40 CFR 136 specifies that samples analyzed for gross alpha and beta activity are subject to a holding time of 6 months, that requirement should be specified as applicable holding time criteria, replacing the criteria above, for gross alpha and beta measurements.

Physical characteristics and matrix influences must also be considered when setting holding times. These characteristics must be considered when planning for validation implementation. The following table presents commonly-analyzed radionuclides on the DOE Complex that have characteristics affecting holding time establishment decisions:

Table 1.1 Physical and Matrix-related Characteristics

Nuclide	Physical or Matrix-related Characteristic
Anionic	Volatile when placed in acid solution
^{3}H	Volatile
^{131}I	Volatile
^{99}Tc	Volatile

The following table presents commonly-analyzed radionuclides on the DOE Complex particularly susceptible to holding time and RDL exceedences due to short half-life:

Table 1.2 Commonly-Analyzed Short Half-Life radionuclides

Nuclide	T *
^{131}I	8.04 days
^{222}Rn	3.82 days
^{210}Po	138 days
^{89}Sr	50.5 days

*Source: Kocher, D. C. Radioactive Decay Data Tables. DOE/TIC-11026.

Data Verifier

a. Verification

- Check sample data and quality indicator sample data for instances of MDA>RDL. Note exceedences of the RDL in the verification report.

- Turn-around time is evaluated as **strictly a contractual issue**. An agreement should be established between the client and the laboratory concerning turn-around times for reported data. If turn-around times are not satisfied, a contractual mechanism should provide for appropriate action.

4. Sample Preservation

When appropriate, or in the absence of known preservation techniques, the preservatives and container types listed in the table 5.3 should be used for aqueous samples. The preservatives listed in this table are applicable to aqueous matrices only. Solid samples (e.g., soils, sediments, sludges) are not amenable to chemical preservation. It is recommended that samples of any matrix not be allowed to be stored in high temperature areas so as to prevent potential loss of volatile radionuclides.

Table 4.1 Preservation and Container Requirements

Nuclide or Group	Preservative	Container
Gross Alpha or Beta	Concentrated HCl or HNO_3 to pH= 2	Plastic or Glass
^{226}Ra or ^{228}Ra	Concentrated HCl or HNO_3 to pH= 2	Plastic or Glass
U, Th	Concentrated HCl or HNO_3 to pH= 2	Plastic or Glass
^{89}Sr / ^{90}Sr	Concentrated HCl or HNO_3 to pH= 2	Plastic or Glass
^{131}I	HCl/ 2M $NaHSO_3$	Plastic or Glass
^{3}H	None	Glass
^{99}Tc	Concentrated HCl or HNO_3 to pH= 2	Plastic or Glass
Gamma Emitters	Concentrated HCl or HNO_3 to pH 2	Plastic or Glass

Data Verifier

a. Verification

Check sample documentation for use of proper preservative and container types. Note occurrences of improper preservative and/or container type in the verification report.

b. Validation

If samples have not been preserved properly in the field or have been stored in an improper container, qualify those sample results < MDA "UJ." Sample results =MDA may not require qualification; however "J" may be placed if necessary.

If samples with radionuclides amenable to preservative with acid have not been acidified in the field, but have been acidified in the laboratory prior to subsampling, qualification may not be necessary. The matrix and container type will not affect the radioactive characteristics of the radionuclides in the sample. For this reason, neglecting to acidify samples prior to shipment to a laboratory should not necessarily result in qualification. However, as radionuclides will adhere to the container walls, acidification (of aqueous samples) either during the sampling event or at the laboratory prior to subsampling is critical to ensure that all radioactive components are in solution, and the representativeness of the sample is maintained.

Data Validator

Qualify sample results < MDA which have not been preserved properly or have been stored improperly UJ. Place J on sample results =MDA only if it is suspected that improper preservation or container type have had the effect of affecting the presence of the radionuclide in the sample.

5. Instrument Calibration

a. Scope

It is outside the scope of this document to prescribe calibration requirements for the laboratory. This section provides an overview of general requirements for initial calibration and calibration verification. Calibration requirements should be found in the analytical laboratory contract and methods.

b. Mechanism for Initial Calibration Evaluation

All calibration data are essential for project records from the standpoint of defending the conditions under which samples were analyzed; however, evaluation of initial calibration data should not involve verification and validation with each reporting batch.

To provide a mechanism for initial calibration evaluation at the project-level, the following approach should be taken.

1. Laboratory Statements of Work must incorporate calibration definitions, frequency, and quality control criteria.

2. Upon award of a contract, the laboratory should provide to the project all initial calibration data for all detector systems to be used under the contract as a one-time deliverable (calibration verification data shall be submitted per data package, as described in 4a below). At this time, the control status of initial calibration can be evaluated. The contract must require that the laboratory update the project with changes in calibration status. Initial calibration tracking numbers (i.e., the unique identification of a detector) for initial calibration must be included in all reporting batches for reference purposes.

3. If the necessary deliverables for initial calibration cannot be provided by the laboratory, two potential options exist:

   (a) if missing deliverables do not significantly affect the ability to evaluate sample data, the project may accept the initial calibration data

   (b) if missing deliverables either present a significant nonconformance for project records retention or preclude the ability to evaluate sample data, the project may request the laboratory to perform a new initial calibration in accordance with the deliverables in this section.

c. General Technical Specifications for Initial Calibration

The following technical specifications apply to all initial calibrations and calibration verifications, independent of counting instrumentation category.

1. Check Source Statistics

   The instrument calibration sources should provide adequate counting statistics (less than 1% Poisson statistic uncertainty) over the time period for which the source is to be counted. However, the source shall not be so radioactive as to cause (1) pulse pileups, (2) dead time that is significantly different from that to be expected from routine analysis, or (3) gain shift in the case of pulse height analyzer systems.

2. Radioactive Sources

   Commercially prepared and sealed standards shall not be used after their stated expiration dates, which are based on radionuclide half-life or physical form of the standard, (e.g., sealed source or plated planchet). Standards prepared from certified radioactivity standards at the laboratory or those purchased without expiration dates should be recertified against a certified radioactivity standard source or replaced yearly.

   When using a mixed gamma source, typically two radionuclides decay within 6 months, but the remaining 11 isotopes remain present in measurable quantities. The standard remains usable after significant decay of the two radionuclides, but still is subject to the recertification criteria stated in the above paragraph.

   The standard source(s) used in initial calibration shall be NIST-traceable Standard Reference Materials, or equivalent; however, source(s) used in calibration verification are not required to be NIST-traceable, unless measurements of these sources are directly used in calculation of analytical sample data results.

3. Control Criteria

The scope of this document does not include prescriptive requirements for calibration, however, quality of analytical data is highly dependent on control of the calibration process. To facilitate a framework for defining control of the calibration process, the three following strategies may be incorporated dependant on what instrumentation is being used:

(a) Tolerance charts may be established based on consideration of specific performance characteristics of the instrument and radioanalytical method. The required precision of tolerance charts must never be more restrictive than that of a quality control chart.

(b) Statistical quality control charts may be established based only on a level of confidence considered necessary for statistical quality control.

(c) Fixed limits may be used by consideration of percent deviation from a known value. With some radiochemical methodologies (e.g., alpha and gamma spectroscopy) establishment of tolerance or statistical quality control charts may provide unrealistic precision goals (e.g., 5% relative percent difference, or RPD) may exceed a ±3s control chart limit but still provide adequate instrumental precision). In these cases evaluation of measured values using a percent deviation approach may provide realistic evaluation of detector precision.

4. Establishment of control points

Establishment of control points for use with a tolerance or statistical quality control chart may be approached in two differing strategies, fixed range or moving range.

(a) Fixed range control charts are established by acquiring a predetermined number of points, with associated mean and standard deviation, and comparing subsequent data point acquisitions to those statistical descriptors. This allows for evaluation of instrumental control over time, but may not represent true precision over time.

(b) Moving range control charts are established by acquiring a predetermined number of points, with associated mean and standard deviation, and as subsequent points are acquired, they are included for an up-to-date evaluation of system precision. In using a moving range control chart, only the most recent 20 points are considered in establishing statistical descriptors. The use of moving range control charts allows for real-time evaluation of detector control, but does not allow for evaluation of detector control in relation to initial calibration.

5. Control of Background

   The control limits for check sources and backgrounds (where applicable) shall be established using a minimum of 20 sequentially measured data points. For extended background count periods, a series of at least 10 single background measurements is acceptable. No samples, subject to these specifications may be counted until these warning and control limits have been established.

   Background count time should be equal to or greater than sample count time unless precluded by extended low level sample count times, in which case background count time may be less than sample count time.

6. Geometry

   With all methods of detection, the calibration counting geometry used must be the same as that used with the analytical samples.

7. Background subtraction

   Calibration data must be background-subtracted, whether data is used in generation of efficiencies, cross-talk, or resolution evaluation.

8. Recalibration

   Two potential justifications exist for recalibrating an instrument: (1) according to manufacturers specifications and (2) according to failure in response to a calibration quality control (QC) test.

   Recalibration shall occur within the manufacturers specifications or as defined by laboratory procedures. Recalibration shall occur after major components are changed or repair in such a fashion that there is a potential effect on instrument performance. All defined performance criteria shall be re-performed as discussed in this section, including the creation of new control charts.

   Recalibration is necessary in the event that the instrument/system has malfunctioned and the repaired equipment has responded to a calibration QC test in a fashion that the tolerance level of a control chart has been exceeded, (i.e., the operating or response characteristics of the instrument/system have changed more than the tolerance/control limits permit). Detector calibration is verified according to frequencies that will satisfy contractual criteria, and according to criteria defining the warrant of corrective action.

d. Technical Specifications for Calibration Verification

1. Calibration verification data must be submitted with each reporting batch and will be evaluated at that frequency. If samples within a reporting batch are from separate counting batches, calibration verification documentation must be included for all counting batches.

2. Each reporting batch submitted from the laboratory to the project should contain control charting data related to calibration verification for all detectors used in the analyses of the analytical samples.

3. Calibration verification is performed and monitored with tolerance or QC charts for instrumental parameters specific to each type of detector. If the daily check source count result exceeds the tolerance limits or ±3s control limit, the laboratory should recount the check source to verify the out of control condition. If the recount again exceeds the control limit, the system is considered out of control, and no samples shall be run on that system until it is brought back into control. If the recount is in control a third count shall be done and if in control, analytical sample counting may continue, otherwise no samples shall be analyzed on that system until it is brought back into control. Any samples counted after the last in-control check standard must be recounted except for those where decay has eliminated that radionuclide.

4. If calibration verification data exceed the tolerance limits or the ±3s control limits, reference must be made to quality control sample data in the data package to determine the extent of calibration nonconformance on the counting batch. Exceeding the control limits may not constitute qualification of data; but conversely, excessive control limit exceedence may affect all data in a counting batch, justifying qualification.

5. Initial Calibration

Data Verifier

(a) Verification

For all methods, verify the presence of initial calibration deliverables listed in the appropriate radio analytical SOW or method.

6. Calibration Verification

Data Verifier

(a) Verification

Verify the presence of deliverables listed in the appropriate radioanalytical SOW or method. Contact the laboratory if deliverables are found to be inconsistent with either of these listings.

Data Validation

(b) Validator

Examine the verification report, and place the appropriate reason codes on affected data for non-correctable deliverable deficiencies. Evaluate control charts for out-of-control conditions. Qualify affected data "J" or "R" based on specific conditions in the counting batch.

Evaluate standard source age(s). If standard source(s) have aged greater than the expiration date on the certificate(s), affected sample results should be qualified "J," and qualified "R" using professional judgement.

6. Quality-Indicator Samples

Quality-indicator samples are evaluated during analytical data validation to determine the control of the analytical method and matrix-related effects on sample data. For analytical data validation, quality indicator samples help determine what radioanalytical conditions affect the usability of the data.

The strategy by which quality-indicator samples are evaluated involves an evaluation of whether the difference between expected and measured results is statistically significant when compared to their TPU. The mathematical relationships presented in the following sections are compared to a factor corresponding to a statistical level of confidence. When the relationship exceeds the factor, the two results differ at that statistical level of confidence when compared to their TPU.

The statistical assumption inherent in these tests is that sample results are drawn from normally distributed populations with estimated means and known variances. Factors in the TPU relationship may originate from populations which are not necessarily normally distributed (e.g., counting uncertainty). However, use of sample results and TPU, assuming approximation to the normal distribution, provides a reasonable and appropriate approach to evaluating control of analytical conditions. Presented in this procedure are statistical decision-making levels at 95% and 99% levels of confidence (decision-making factors are 1.96 and 2.58, respectively). Projects may choose other levels of confidence and decision-making factors based on project DQOs, with the realization that qualification decisions made through validation will be at differing levels of confidence and conservatism. The following table provides examples of these decision-making factors which are applied as decision-making tools through this procedure:

Table 6.1 Examples of Confidence Levels for Qualification Decision-Making

Confidence Level, %	Decision-making factor	Decision-marking level
50	0.68	More conservative
67	1.00	
75	1.15	
90	1.645	
95	1.960	
99	2.575	Less conservative

Adapted from: Taylor, J.K. Quality Assurance of Chemical Measurements. Lewis Publishers, 1997.

Listed in this section are guidance for qualification for single quality-indicator samples being outside control criteria based on a 95% and 99% level of confidence. Analytical samples should not be rejected based on a singular quality control sample. Effects of other QC sample deficiencies must be considered to evaluate whether conditions are such to justify rejection of data. Attachment H provides analytical decision-making guidance for situations where multiple quality deficiencies are encountered.

The tests in this section are meaningful only if the radioanalytical method functions properly. If a method is deemed seriously out of control, the tests in this section are not appropriate, and no further validation needs to be done; all results may be considered unusable.

Analytical samples and the respective quality indicator samples may be counted on differing detectors within the same detector system, provided that calibration is within appropriate control limits. However, quality indicator samples counted on differing detector systems from the analytical samples may not provide meaningful data, and qualification may be appropriate.

a. Total Propagated Uncertainty (TPU)

The tests presented in this section rely heavily upon evaluation of uncertainty associated with radioanalytical results. The random factor in the TPU relationship is the counting uncertainty; the remaining terms comprise the systematic factors. Many laboratories choose to report uncertainties separately as total random and total systematic. These factors are acceptable to use in the tests in this section providing that the components of the uncertainties are recognized. A laboratory should include in their TPU equation, counting uncertainty, efficiency uncertainty, and recovery uncertainty. Other terms can be propagated individually or a collective term may be used. The collective term being a combination of the systematic uncertainty summed with any other relatively minor, random individual uncertainties. The laboratory should explicitly show which terms were individually propagated and which were bundled together. In all cases, significant uncertainties should be propagated for the laboratory TPU calculation.

In the event that not all the requested uncertainties are available, the magnitude of TPU must be evaluated considering which components are the dominant factors in the relationship. At relatively low count rates, the random components will likely be the dominant factors; and at high rates, systematic components may be dominant.

b. Standard Traceability

Standards used in the preparation of quality control samples (laboratory control samples, matrix spikes) or sample-specific spikes (tracers or carriers) shall be shown to be traceable to a reliable source (e.g., NIST, IAEA).

1. Verification

Data Verifier

Verify the identity of standard(s) used in the relevant quality-indicator sample preparation and sample-specific spiking by tracing the standard control number from the certificate to sample preparation documentation. If a standard is not traceable, note the occurrence in the verification report.

2. Validation

Data Validator

Examine the verification report. Place reason code E02" on sample data affected by non-traceable standards. The reason code must be placed on all sample data related to non-traceable quality-indicator samples.

c. Laboratory Control Sample (LCS)

The purpose of a LCS is to monitor the accuracy of the preparation and analysis of the analytical samples, provided that LCS is fully homogenized prior to preparation and analysis. The LCS should be the same matrix type as the analytical samples (e.g., water, soil). Exceptions should be made in cases of novel matrices (e.g., sludge, oil, biota). The spike in the LCS should be of a level near that of the analytical samples, or contractually specified at a predetermined level (e.g., 10 times the MDA may be appropriate).

Three types of LCS material have been identified as being appropriate to evaluate laboratory process accuracy (please see definitions section for further detail):

- Traceable Reference Material (TRM)
- Standard Reference Material (SRM)
- Well Characterized Reference Material (WCRM)

1. Verification

Data Verifier

Verify that one LCS was prepared at the same time with the analytical samples, was of the same matrix as the analytical samples, and has been analyzed with an analytical batch of up to 20 analytical samples. If these criteria were not met, state the occurrence as a non-correctable problem in the verification report.

2. Validation

Data Validator

Place the reason code "L05" on affected sample results if LCS data are not reported.

LCS – Test for bias

Use the following mathematical relationship to test the null hypothesis that the expected and measured results of the LCS do not differ significantly when compared to their respective TPU.

$$\frac{LCS_{meas.} - LCS_{\exp.}}{\sqrt{(TPU_{meas.})^2 + (TPU_{\exp.})^2}}$$

LCS_{meas} = Measured LCS result
$LCS_{exp.}$ = Expected result of LCS
$TPU_{meas.}$ = 1s Total Propagated Uncertainty of measured result
$TPU_{exp.}$ = 1s Total Propagated Uncertainty of expected result

Qualify sample results = MDA "J" if the normalized absolute difference is between 1.96 and 2.58, as the conclusion is reached that the spiked and expected results differ at the 5% level of significance. Qualify results < MDA "UJ" only if the normalized absolute difference shows a negative bias. If the normalized absolute difference is greater than 2.58 or less than -2.58, consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R" as at the 1% level of significance, the conclusion is reached that the spiked and expected results are different enough to indicate a significant positive or negative bias. If multiple quality deficiencies are encountered qualify using the guidance provided in Attachment H.

d. Laboratory Duplicate

The purpose of a laboratory duplicate is to monitor the precision of the analytical method, provided the sample is fully homogenized prior to preparation and analysis. The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. To provide for relevancy of laboratory duplicate data, the sample chosen should have measurable activity (i.e., > MDA and 2s counting uncertainty); however, the test provided in this section may be performed on results < MDA or 2s counting uncertainty. In analytical methods where no sample preparation or separation is performed (e.g., gamma spectrometry), a sample recount may be performed in lieu of a laboratory duplicate, although qualification under these conditions should be based on instrumental performance, as most gamma spectrometry entails minimal sample preparation.

1. Verification

Data Verifier

Verify that one laboratory duplicate was prepared at the same time with the analytical samples, was of the same matrix as the analytical samples, and has been analyzed with a preparation batch of up to 20 analytical samples. If their criteria were not met, state this as a non-correctable problem in the verification report.

2. Validation

Data Validator

If laboratory duplicate data are not reported for a sample(s), place the reason code "D04" on the sample results for the affected preparation batch.

Duplicate - Statistical test for difference between sample and duplicate

Use the following mathematical relationship to test the null hypothesis that the expected and measured results of the laboratory duplicate do not differ significantly when compared to their respective TPU.

$$\frac{S - D}{\sqrt{(TPU_s)^2 + (TPU_D)^2}}$$

S = Sample result
D = Laboratory duplicate result
TPU_s = 1s Total Propagated Uncertainty of the sample
TPU_D = 1s Total Propagated Uncertainty of the duplicate.

If the sample or laboratory duplicate results are less than their respective MDA, the results may still be used in this relationship to determine precision.

Qualify sample results = MDA "J" if the normalized absolute difference is greater than 1.96, as the conclusion is reached that the spiked and expected results differ at the 5% level of significance. If multiple quality deficiencies are encountered qualify using the guidance provided in Attachment H.

e. Matrix Spike (MS)

The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation, provided that samples are fully homogenized prior to preparation and analysis. The matrix spike is a split of a field-originating analytical sample in which one half of the split is spiked with a known amount of radionuclide of interest prior to sample preparation.

Due to difficulties in homogenization of solid samples for gamma analyses, a matrix spike may not present useful information. Matrix spikes may present useful information for aqueous samples; but should not be used for energies <100keV.

Matrix spikes may not be required for methods where a carrier or tracer is used, provided that the tracer chosen is chemically similar to the radionuclide of interest. Matrix effects will be detected through tracer recovery; however, difficulty may be experienced in ascertaining that poor recovery is due to matrix effect or through losses in separation.

1. Verification

Data Verifier

Verify that one matrix spike was prepared at the same time with the analytical samples, was of the same matrix as the analytical samples, and has been analyzed with a preparation batch of up to 20 analytical samples. If this frequency was not met, state this as a non-correctable problem.

2. Validation

Data Validator

Place the reason code "M05" on the sample results for the affected preparation batch if matrix spike data are not reported.

Matrix Spike - Test for matrix-induced bias

Use the following mathematical relationship to test the null hypothesis that the expected and measured results of the MS do not differ significantly when compared to their respective TPU.

$$\frac{SSR - SR - ER}{\sqrt{(TPU_{SSR})^2 + (TPU_{SR})^2 + (TPU_{ER})^2}}$$

SSR = Spiked Sample Result
SR = Sample Result (unspiked)
ER = Expected Result
TPU_{SSR} = 1s Total Propagated Uncertainty of Spiked Sample Result
TPU_{SR} = 1s Total Propagated Uncertainty of Sample Result
TPU_{ER} = 1s Total Propagated Uncertainty of Expected Result

Qualify sample results = MDA "J" if the normalized absolute difference is between 1.96 and 2.58, as the conclusion is reached that the spiked and expected results differ at the 5% level of significance. Qualify results < MDA "UJ" only if the normalized absolute difference shows a negative bias. If the normalized absolute difference is greater than 2.58 or less than -2.58, consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R" as at the 1% level of significance, the conclusion is reached that the spiked and expected results are different enough to indicate a significant positive or negative bias. If multiple quality deficiencies are encountered qualify using the guidance provided in Attachment H.

Consideration should be given to the similarity in matrix type among samples in the preparation batch batches. If the matrices differ notably (particularly in soil particle size) qualification may be placed on only the sample associated with the matrix spike. If matrices do not differ notably, qualification may be placed on all samples in the preparation batch. If multiple quality deficiencies are encountered, qualify using the guidance provided in Attachment H.

f. Method Blank

The purpose of a method blank is to monitor the presence of contamination of the analyte of interest in the sample preparation and analysis processes. The method blank is a laboratory-generated sample of the same matrix as the analytical samples, but in absence of the analyte of interest.

1. Verification

Data Verifier

Verify that all method blank activities are less than their MDA and 2s counting uncertainty. If method blank activity is greater than the MDA or 2s counting uncertainty, note this in the verification report.

Verify that one method blank was prepared at the same time with the analytical samples, was of the same matrix as the analytical samples, and has been analyzed at a frequency of one per preparation batch of up to 20 analytical samples. If these criteria were not met, state this as a non-correctable problem in the verification report.

2. Validation

Data Validator

Place the reason code B07" on sample results for which a method blank is not reported.

If either of the following conditions are met, no actions outlined in this section need to be performed:

- method blank result is less than its MDA or less than its 2s counting uncertainty, or
- method blank result is greater than its MDA with the sample result less than its MDA.

Validation of the method blank tests whether blank contamination is of a level which may be indicative of systematic contamination through sample preparation.

If both the method blank and sample activity are greater than their respective MDA or 2s counting uncertainty, the following mathematical test shall be used in determining the effect of blank contamination on sample results. Use the mathematical relationship below to test the null hypothesis that the sample and the method blank do not differ significantly when compared to their respective TPU.

This test may be used as long as the method blank is reported in terms of activity per unit weight or volume, consistent with the sample results. If sample results are reported as method blank subtracted, the unsubtracted result must be used in the relation below.

$$\frac{|S - B|}{\sqrt{(TPU_S)^2 + (TPU_B)^2}}$$

S = Sample result
B = Method blank result
TPU_s = 1s Total Propagated Uncertainty of Sample
TPU_B = 1s Total Propagated Uncertainty of Method Blank

Qualify sample results = MDA "J," if the normalized absolute difference is between 1.96 and 2.58 as the sample and method blank differ at the 5 % level of significance (sample results < MDA do not require qualification). If the normalized absolute difference is between 0 and 1.96, consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R," the conclusion is reached that the method blank and sample results differ at the 1% level of significance. If multiple quality deficiencies are encountered, qualify using the guidance provided in Attachment H.

7. Elevated Uncertainty

The statistical tests described in section F tend to qualify data less conservatively at progressively increasing levels of TPU compared to the sample result. At some level of uncertainty, sample result qualification may be needed to alert the data user that the sample result may be too uncertain to use for an intended purpose. The following test qualifies data as a function of TPU (although counting uncertainty may be used instead of TPU) as a percentage of the sample result. The percentage level may be determined by the individual project through the DQO process in deciding the acceptable level of uncertainty the data user is willing to accept. The percentage in the test below has been determined through inspection of existing radiochemical results.

a. Validation

Data Validator

Examine sample results and total propagated uncertainties for each result. If the TPU is = 80% of the sample result, qualify the affected sample results "J."

8. Chemical Yield – Tracers and Carriers

Tracers and carriers are used in radiochemical separations methods to provide evaluation of chemical separation. Chemical yield is evaluated through the recovery of chemical species spiked into samples. Yield is evaluated radiometrically with a tracer and gravimetrically with a carrier. Each sample is spiked with either a carrier or tracer, and sample results are adjusted for yields greater or less than 100%.
Generally, a low yield is indicative of losses of tracer and radionuclide of

interest through sample separation, and recoveries greater than expected (> 100%) are indicative of instrumental problems, contamination, or presence in sample; tracers and carriers are not expected to be recovered at levels greater than spiked.

a. Verification

Data Verifier

Verify that for applicable analyses, one carrier or tracer recovery is reported for each sample. If a carrier or tracer percent recovery is not reported for each sample, contact the laboratory for submittal of this data. If the data can not be provided, state this as a non-correctable problem in the verification report.

As yield decreases, the MDA may elevate to a point at which the RDL is exceeded, and analytical results are contractually noncompliant. If the laboratory has not initiated corrective action, for samples in which the MDA exceeds the RDL, the project may choose to contact the laboratory for sample re-work. If re-work is not feasible, indicate the noncompliant data in the verification report.

b. Validation

Data Validator

Place the reason code "Y04" on the sample result if yield is not reported for a sample(s). Yield is validated based on percent recovery of the spiked nuclide. Low yield may be indicative of increased uncertainty in the sample result. Criteria for qualification should be based on what magnitude of correction has been applied to the sample result (e.g., 20% recovery implies a sample result correction of 5), although a point of debate exists concerning usability of radionuclide data with yields near 0%. Yield criteria may also be established from existing sample yield data from previous sampling at the site, if this data are available.

Qualify sample results with yield outside accepted limits J and R as appropriate; however, sample results should not be qualified based on yield alone. Sample yield should be evaluated in reference to chemical yield of quality-indicator samples. If yield is generally low throughout the preparation batch, but recoveries of target radionuclides in the LCS are acceptable, data may be accepted without qualification; however, if quality control sample yield is generally low, sample results with low yield may need qualification.

9. Required Detection Limits (RDL)

a. Verification

Data Verifier

Verify that MDA<RDL for all radionuclides; note occurrences of RDL exceedence in the verification report **only** for those samples for which sample results are reported at levels < MDA.

b. Validation

Data Validator

Verify that MDA<RDL for all radionuclides; note occurrences of RDL exceedence in the verification report **only** for those samples for which sample results are reported at levels < MDA.

10. Nuclide Identification and Quantitation

a. Test for Detection

Data Validator

1. Validation

(a) Evaluate whether a sample result is greater than its MDA.
(b) Evaluate whether the sample result is greater than its 2s counting uncertainty.

If the sample result is less than its MDA or less than its 2s counting uncertainty, qualify the sample result "U."

If the sample result is less than its MDA (assuming that the MDA is reported at the 95% confidence level) the data user is accepting the probability of 5% of a false negative result. If the sample result is less than its 2s counting uncertainty, at the 95% confidence level, the radionuclide result is not significantly different from zero.

In placing the "U" qualifier, it should be realized that RDL exceedence may occur due to excessive radionuclide disintegration. Also, special attention must be paid to instances where nominal conditions are not followed (i.e., sample aliquot size, count time). Sample results which are less than their MDA, but greater than their 2s counting uncertainty may have been counted for a period of time long enough to be considered detected. Sample results which are greater than the MDA, but less than the 2s counting uncertainty may not have been counted for a period of time long enough to be considered detected.

b. Negative Results

Data Validator

1. Validation

Place qualifiers using professional judgement due to occurrence of negative results. The rationale for qualifier placement must be fully described in the validation report.

Negative results that have absolute values greater than their 2s counting uncertainty are an indication that the instrument background has shifted. The implication of unstable background is a possible negative bias in the sample result. Qualification due to very negative sample results must take into account evaluation of calibration verification background and quality-indicator samples that may indicate a bias.

11. Sample Result Recalculation

The accuracy and consistency of sample result calculation by the laboratory can be addressed through two different techniques. The application of each strategy depends on the laboratory's ability to minimize transcription during reporting, and how familiar the project is with the performance of the laboratory.

a. If sample results are produced primarily through software processing and minimal transcription is performed in the laboratory, the data system(s) can be evaluated during an audit or surveillance by performing two different tests on the software: (1) supply the data system a consistent set of input designed to provide a consistent set of output, and (2) supply the data system a set of nonconforming data to test the error detection routines. An evaluation of the laboratory's software configuration control and security is also necessary. If reporting software has been developed for customized use in the laboratory, additional confidence is gained through the laboratory's adherence to a software quality assurance standard and through maintenance of a software verification and validation plan. Through this technique, a high level of confidence can be gained in the laboratory's reporting techniques and will result in a minimal need for manual recalculation of sample results. Recommended surveillance considerations for gamma algorithms are included in Attachment F.

b. If the laboratory has a high rate of manual transcription in generation of sample results, the project may choose to manually recalculate sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contacting the laboratory may be necessary to resolve the problem. Qualifiers should be placed using professional judgement.

Activity and TPU equations provided in Attachment G are useful for providing the basic structure for calculating radioanalytical results. Modifications to the equations may be needed in method-specific cases. All calculations used should be contractually agreed upon between the project and the laboratory prior to sample analysis, and iterated in the laboratory SOW.

12. Instrument-Specific Sample Considerations

a. Sample Analytical parameters for Gas Proportional Counting

1. Validation

Data Validator

Equations to approximate a relationship between standard weight and activity are detector-specific. They are developed from the mass attenuation curves by the laboratory and are applied to calculate sample-specific efficiencies. If recalculating sample results, efficiencies used must be sample-specific.

A representative sample aliquot must be chosen to ensure the dissolved solid content of the sample falls within the mass range of the appropriate curve.

Qualify sample results "J" for which aliquot weight is outside the mass attenuation curve if not reanalyzed with a smaller aliquot.

b. Spectral Interpretation (alpha and gamma spectrometry)

1. Gamma Spectrometry

Identification of the radionuclides associated with the measured gamma-ray energies and intensities is largely performed by gamma-ray spectral-analysis algorithms. These algorithms automatically locate peaks in spectra by applying a user-definable set of criteria. Based upon this set of criteria, the peaks in a spectrum are distinguished from the Compton edges, backscatter peaks, and the numerous features created by the background radiation and detection process. The algorithms automatically calculate the peak areas and correct them for the system's detection efficiency as a function of gamma energy. The identification of the radionuclides that produced the peaks is accomplished by comparison to the information in a customizable radionuclide analysis library. For all peaks identified in a spectrum (that corresponds to a radionuclide in the analysis library), the algorithms calculate the activities of the identified radionuclides in the sample. However, the use of peak search, identification, and quantification algorithms does not preclude the need for laboratory data review by an experienced spectroscopist.

The resulting quality of the gamma spectrometry data will largely depend upon the ability of the spectroscopist to establish a set of analysis criteria to be used by the algorithms with perform the location, identification and quantification of features within spectra. The quality of this process is greatly influenced by the spectral library. For this reason, it is recommended that the library originate from a reliable source and software be controlled with respect to changes in identification criteria. Multiple libraries are often constructed based upon expected interferences and the level of activity in the samples. The multiple libraries reduce the number of unidentified peaks due to summation events and interferences within the sample matrix. The goal of reducing the number of unidentified peaks within a spectrum, while not compromising the analysis routines by the inclusion of extremely low abundance branching probabilities, helps limit the uncertainty of detection of those radionuclides which emit more than one photon.

The data package should include both identified and unidentified peaks. This will support the evaluation of instrumental system control.

2. Validation of Gamma Spectra

The assurance of radionuclide identification and quantitation by properly functioning algorithms, through reasonable resolution criteria, and by an evaluation by an experienced radiochemist will provide a level of certainty in the quality of the data. Validation of data produced under these conditions should be minimal, (i.e., a thorough review of all spectra including radionuclide identification and recalculation of sample results) should not be necessary. A recommendation is to browse sample-specific spectra for changes in energies positions of target radionuclides and for significant peak overlap.

Radiochemical data produced under substandard or unknown conditions for identification and quantification warrant a closer inspection. Recommended criteria for evaluating gamma spectra in relation to radionuclide identification and resolution are as follows:

(a) Where more than one isotope of a single Naturally Occurring Radioactive Material (NORM) series are reported, reported results for that series should demonstrate secular equilibrium.
(b) For soil samples, two peaks that are almost always observed are the 511 keV annihilation peak and the 1460 keV peak of ^{40}K. The appearance of these peaks at the respective energy, and the respective peak shape should be checked.
(c) If ^{40}K is quantitated in the analysis, the reported value should be checked against the activity expected in site's soil (if those data are available).
(d) For nuclides identified and/or quantitated with two or more gamma energies (i.e., 1173 and 1332 keV for ^{60}Co) the count rate at each energy can be observed to ensure that the count rate at each respective energy is reasonable to confirm the presence of the isotope.
(e) Where isotopes are verified by software using several energies, ensure isotopes are not disqualified by low abundance energies when counting times limit detection for those energies.

3. Alpha Spectra

As with gamma spectrometry, the alpha spectrometry can require the use of algorithms for the location, identification, and quantification of peaks created by radionuclides with spectra. While many of the analysis algorithms are the same for alpha spectrometry as gamma spectrometry, there are fundamental differences which effect the validation of data. These differences which are inherent to the mode of the decay, sample preparation techniques, and phenomena associated to the detection and quantification require validation. As with gamma spectrometry, if algorithms and operational criteria are employed by the laboratory, then they should be reviewed by a competent spectroscopist and/or radiochemist.

Alpha spectrometry has limitations due to the alpha particles ability to transfer energy at an extremely high rate. This phenomenon is responsible for alpha particles being significantly attenuated by the sample matter, the mass of the radionuclide itself, and the molecules of air between the detector and the sample. If the mass of the samples is not reduced or the radionuclides are present in appreciable amounts, then likelihood of receiving meaningful data is reduced due to attenuation. Therefore, samples for alpha spectrometry frequently require extensive radiochemical preparations, with the sample being placed in vacuums and in close proximity to the detectors. Typically, these separation/preparation techniques isolate one or two target nuclides, thus reducing the possible attenuation which occurs at high masses.

This separation process presents the possibility that contaminating elements could break through the chemical separation process. If chemical breakthrough were to occur, these interferences could erroneously be incorporated into the quantification of the radioisotope of interest.

Unlike gamma spectrometry, the spectra resulting from properly prepared alpha sources/samples are relatively simple, since the peaks are near Gaussian, the background is very low, and detector efficiency is almost uniform for the 3.0 MeV to 6.5 MeV energy range. Frequently, the use of regions of interest (ROI) are integrated to determine gross activity which can be adjusted for background and then corrected for chemical recovery, decay, aliquot, and detection efficiency to produce concentration. A ROI peak report, including all information needed to manually recalculate results, is useful to evaluate data, as well as the spectra itself.

4. Validation of Alpha Spectra

Alpha spectra are not generated through the application of algorithms; consequently the approach to validation differs from that of gamma spectrometry. The following two tests should be performed on alpha spectral data:

(a) target peaks should be in the energy range of interest, and
(b) peak tailing should not significantly overlap peaks at lower energies.

Professional judgement should be used in qualification of sample data based on spectral interferences or peak misidentification. Communication with the laboratory is essential to obtaining reliable identification/quantitation of suspect radionuclides

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23. Upgrading Environmental Radiation Data, Health Physics Society Committee Report HPSR-1 (1980), EPA 520/1-80-012, U.S. Environmental Protection Agency.

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RECORDS

1. General Records

Records arising from the actions in this document are as follows:

- The Implementation Worksheet;
- The verification report;
- The validation report; and
- Copies of qualified, or unqualified results reports.

These records become part of the analytical data package and must be controlled through a records management system.

2. Quality Assurance Records

This procedure is considered a Quality Assurance Record and, as such, a record copy will be provided to the appropriate document management center (DMC) for retention according to the minimum schedule identified by DMC personnel.

SOURCE DOCUMENTS

- "Analytical Data Validation Plans for Environmental Restoration Projects," Environmental Restoration Procedure ERWM/ER-P2200.015 (Tracking No.), Lockheed Martin Energy Systems, Inc., Oak Ridge, Tennessee (DRAFT).

- *Environmental Measurements Data Management Plan for the Environmental Restoration Program*, ES/ER/TM-88 R1, Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee.

Attachment A
DEFINITION/ACRONYMS

Activity: Activity is defined as the number of spontaneous nuclear transformations which occur in a quantity of a radioactive nuclide per unit time.

Affected Sample Result: A sample result is considered to be affected when it is significantly influenced by a quality deficiency, and is qualified, accordingly, through analytical data validation.

Analytical Data Validation: Analytical data validation is a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Analytical Data Verification: Analytical data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract which is performed by either the data generator or by an entity external to the data generator.

Calibration Verification: Calibration verification, as described in this procedure, is defined as a periodic evaluation of instrument standardization established during initial calibration. Using tolerance or statistical control charts, calibration verification can alert the instrument user of the occurrence of out-of-control instrumental conditions.

Carrier: A carrier is a stable element/compound, introduced into the sample preparation/analysis process, that will behave chemically similar to the analyte isotope(s). It is by virtue of this chemical similarity that the carrier will "carry" the analyte isotope(s) through the sample preparation/analysis process. The amount of the carrier recovered at the end of the analysis compared to that added initially is always used in the calculation of the final result.

Certified Radioactivity Standard Source: A certified radioactivity standard source is a calibrated radioactivity source, with stated accuracy, whose calibration is certified by the source supplier as traceable to a known originator.

Chain of Custody: The history of the transfer of samples from the time of sample acquisition through archival and disposal of samples. Chain-of-Custody documentation is required as evidence of sample integrity.

Correctable Problem: Correctable problems are deficiencies within data packages which may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation. Correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems revealed during validation are those deficiencies with analyses that can be solved by either a second preparation and/or analysis of a sample.

Counting Batch: A counting batch is a group of sample aliquots analyzed together on the same instrument detector system.

Counting uncertainty: Counting uncertainty, as described in this procedure, is defined as the statistical sample standard deviation, which is an approximation of the population standard deviation, and is numerically defined as the square root of the number of counts obtained from a detector. This relationship holds true, provided that the distribution that the counts follows the Poisson distribution. Units for counting uncertainty are the same as for the reported result, Minimum Detectable Activity (MDA), and Total Propagated Uncertainty (TPU).

Data Quality Objectives (DQOs): DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO Process which specify the study objectives, domain, limitations, the most appropriate type of data to collect, and specify the levels of decision error that will be acceptable for the decision.

Data Quality Objectives Process: The DQO Process is a quality management tool based on the scientific method and developed by the USEPA to facilitate the planning of environmental data collection activities. The DQO Process enables planners to focus their planning efforts by specifying the use of the data (the decision), the decision criteria (action level), and the decision maker's acceptable decision error rates.

Holding Time: Holding time, as described in this procedure, is defined as the period of time between sample collection and sample activity determination.

Initial Calibration: Initial calibration, as described in this procedure, is defined as the standardization of an instrument used in radioactivity detection against a traceable radioactive source or standard of known identity and quantity. This standardization prevails until such time as analytical conditions are deemed out of acceptable tolerance or statistical control limits.

Laboratory Duplicate: The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

Lower Limit of Detection (LLD): Lower Limit of Detection is defined as the amount of analyte material that has a 95% chance of being detected when the decision that some amount of analyte is present is made when a signal occurs at or above the decision level. It has the same meaning as minimum detectable activity (MDA), which is preferred terminology

Matrix Spike: The matrix spike is a split of a field-originating analytical sample in which one half of the split is spiked with a known amount of radionuclide of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation

Minimum Detectable Activity (MDA): The MDA is the amount of a radionuclide, which if present in a sample, would be detected with a probability of non-detection while accepting a probability of erroneously detecting that radionuclide in an appropriate blank sample. For this procedure, the probabilities are both set at 0.05. As defined here, the MDA applies to the nominal concept of detection, (i.e., specific to an instrument, radioanalytical method and typical sample type).

Non-Correctable Problem: Non-correctable problems are deficiencies within data package which preclude the evaluation of data quality by predefined criteria. Non-correctable problems may be revealed during both data verification and data validation.

Preparation Batch: A preparation batch is a group of sample aliquots prepared together at the same time using the same method and related to the same quality control samples.
Quality Control Chart: For purposes of this procedure, a quality control chart is a representation of a series of measured values, with statistical descriptors, (i.e., mean and standard deviation limits), and is used to determine if the response of the instrument has changed statistically. The magnitude of the statistical response change may or may not be significant when compared to the required precision and accuracy criteria for the overall analytical technique.

Quality-indicator Sample: Quality-indicator samples are those samples made ready in the laboratory which provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

Reporting Batch: A reporting batch is a group of sample results reported together in a single data package. The reporting batch may be comprised of samples prepared and analyzed together in the same preparation and counting batches or samples prepared and analyzed in different preparation and counting batches.

Required Detection Limit (RDL): The RDL is a contractually specified detection limit which, under typical analytical circumstances, should be achievable.

Sample Result: A sample result, as described in this procedure, is a numeric denotation of the concentration, amount or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

Standard Reference Material (SRM): A SRM is a material or substance of which one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Statement of Work (SOW): The validation SOW is a document prepared by the ER project to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

Tolerance Chart: For purposes of this procedure, a tolerance chart is a representation of measured values with statistical descriptors, (i.e., mean and standard deviation limits) and is based upon maintaining a change of instrument response to a tolerance level judged acceptable to meet overall quality requirements for the technique; a tolerance level should never be more restrictive than what is statistically possible.

Total Propagated Uncertainty (TPU): TPU is the addition of the square root of the sum of the squares of random components of the individual uncertainties, plus the magnitude of the estimated individual systematic relative uncertainties. TPU may include uncertainties introduced through field sampling and analytical laboratory procedures. For the purposes of this procedure, TPU includes only those random and systematic uncertainties associated only with laboratory preparation and analysis. Refer to Appendix D for a full description of TPU.

$$TPU = \sqrt{\Sigma R_i^2 + \Sigma S_j^2}$$

R = random components of individual relative uncertainties
s = magnitude of the estimated individual systematic relative uncertainties

Traceable Reference Material (TRM): A TRM is a NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).

Tracer: A tracer is a radioactive isotope, introduced into the sample preparation/analysis process, that will behave chemically similar to the analyte isotope(s). The tracer isotope is of the same element as the analyte isotope(s) except where the decay mode, half-life, or availability dictate the use of the isotope of a different element. The activity of tracer detected at the end of the analysis compared to that added initially is used in the calculation of the final result.

Turn-around Time: Turn-around time is contractually specified as the amount of time which elapses between laboratory receipt of the raw samples and subsequent data receipt by the client.

Validation Qualifier: A qualifier is an alphabetic character physically or electronically associated with a discrete sample result during validation due to a data quality deficiency, which provides guidance in data usability.

Validation Reason Code: A validation reason code is an alphanumeric character physically or electronically associated with a discrete sample result during validation, which provides maps to a reason for placement of the validation qualifier or, or to a noncorrectable deliverable deficiency in absence of a validation qualifier.

Well Characterized Reference Material (WCRM): The WCRM may be derived from a field sample which has been well characterized through multiple analyses, providing a high level of confidence of the activity level in the sample. The WCRM may be submitted

Attachment B
RADIOCHEMICAL DATA VERIFICATION REPORT

(The actual form is provided on the next page and may be printed and used directly.)

Radiochemical Data Validation Report

Reporting Batch #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW #: _____

This checklist is to be used for verification of radiochemical data packages, consistent with the procedure ES-A-2209, Rev. 0.

Verified by:_____ Date:_____

	Y	N	n/a
Custody of Samples			
1. Are samples traceable through inspection of signature records on field and laboratory chains of custody?	___	___	
Action: For any samples not traceable upon inspection of chains of custody, indicate the identities below and where the break in traceability occurred.			
Turn-Around Times			
2. Has contractual turn-around time been met for all samples?	___	___	
3. Have all samples been preserved correctly?	___	___	
4. Are all sample MDC # RDL?	___	___	
Standard Traceability			
5. Have certificate(s) been included for the LCS?	___	___	
6. Have certificate(s) been included for the MS?		___	___
7. Standards have not aged greater than 5 half lives.	___	___	
Calibration Verification			
8. Have appropriate control charts been included?	___	___	
Laboratory Control Sample (LCS)			
9. Has the LCS been reported with the following parameters?			
a. Measured result of LCS expressed in activity/unit weight or volume.	___	___	
b. Total Propagated Uncertainty for LCS expressed in activity/unit weight or volume.		___	___
c. 2s counting uncertainty for LCS expressed in activity/unit weight or volume	___	___	
c. MDC of LCS expressed in activity/unit weight or volume.	___	___	
d. Expected result of LCS expressed in activity/unit weight or volume.	___	___	
e. Expected counting uncertainty of LCS expressed in activity/unit weight or volume.	___	___	
10. Was LCS prepared with a maximum of 20 analytical samples?	___	___	
11. Was the LCS of the same matrix as the samples?	___	___	
Laboratory Duplicate			
12. Has the Laboratory Duplicate been reported with the following parameters?			
a. Measured result of sample expressed in activity/unit weight or volume	___	___	
b. Measured result of duplicate expressed in activity/unit weight or volume	___	___	
c. MDCs of sample and duplicate expressed in activity/unit weight or volume	___	___	
d. Total Propagated Uncertainty of sample and duplicate expressed in activity/unit weight or volume	___	___	
13. Was the duplicate prepared with a maximum of 20 analytical samples?	___	___	
Matrix Spike (MS)			
14. Has the MS been reported with the following parameters?			
a. Measured result of the unspiked sample in activity/unit weight or volume.	___	___	
b. Measured result of MS expressed in activity/unit weight or volume or volume	___	___	
c. Expected result of the MS expressed in activity/unit weight or volume.	___	___	
d. Expected Total Propagated Uncertainty of the MS expressed in activity/unit weight or volume	___	___	
e. MDCs of unspiked sample and MS expressed in activity/unit weight or volume		___	___
f. Total Propagated Uncertainty of unspiked sample, and MS expressed in activity/unit weight or volume	___	___	
15. Was MS prepared with a maximum of 20 analytical samples?	___	___	

	Y	N	n/a
Method Blank			
16. Has the method blank been reported with the following parameters?			
a. Measured result of method blank expressed in activity/unit weight or volume	___	___	
b. MDC of method blank expressed in activity/unit weight or volume	___	___	
c. Total Propagated Uncertainty of method blank expressed in activity/unit weight or volume	___	___	
d. Was the method blank prepared with up to analytical 20 samples?	___	___	
Chemical Yield - Tracers and Carriers			
17. For methods requiring a tracer or carrier, has a chemical yield been reported for all samples and QC samples?	___	___	
Reporting Verification			
18. Are results, "2s counting uncertainties, and "2s Total Propagated Uncertainty present for all samples that were sent to the laboratory?	___	___	
19. Are any negative results reported?	___	___	

Action:	Indicate non-correctable contract compliance deviations below.

_____

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Attachment C
RADIOCHEMICAL DATA VALIDATION REPORT

(The actual form is provided on the next page and may be printed and used directly.)

Radiochemical Data Validation Report

Reporting Batch #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW #: _____

This checklist is to be used for validation of radiochemical data packages, consistent with the procedure ES-A-2209. This checklist should be used only if the Radiochemical Verification Report has been completed.

Instructions: If criteria are not met in this checklist, follow qualification provided in the qualification ("Q") column accompanied with the corresponding reason code indicated in the code ("C") column. If necessary, additional codes may be placed.

Verified by:_____ Date:_____

1.0 Custody of Samples

Validation Step
Action: For any samples not traceable upon inspection of verification report and chains of custody, indicate the identities below and where the break in traceability occurred. Qualify samples not traceable "R (E01)." Indicate which samples were affected below.
Qualified Sample _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

2.0 Turn-around Times

Validation Step	Y	N	Q	C
1. Do all samples show MDC RDL?			--	Q13
2. Were all samples preserved correctly?			J	H05
Action: Indicate affected samples below.				

Sample	MDC > RDL	Improper Preservative

3.0 Standard Traceability

Validation Step			
Action: If certificates have not been included for the LCS, or MS, place code "E02" on the sample result(s).			
List which certificates were included in the data package.			
Affected Sample	Standard	LCS	MS

4.0 Calibration Verification

Validation Step	Y	N	n/a	Q	C
1. Are efficiencies within the appropriate control criteria?				J/R	C27
2. Are instrument backgrounds within the appropriate control criteria?				J/R	C28
3. Are energies within the appropriate control criteria?				J/R	C29
4. Are appropriate peak resolution within appropriate control criteria?				J/R	C30
Action: With the evaluations presented in this section, qualify affected data "J" unless serious deficiencies warrant rejection. If data are rejected, provide justification below.					

5.0 Laboratory Control Sample (LCS)

Validation Step		Y	N	Q	C
1.	Has at least one LCS been prepared with up to 20 samples?			J	L03
2.	Is the LCS the same matrix as the samples in the reporting batch?			--	L04
3.	Is the normalized difference between the measured and expected values of the LCS within -1.96 and 1.96?			--	--
4.	Does the normalized difference show positive bias between 1.96 and 2.58?			J	L01
5.	Does the normalized difference show positive bias greater than 2.58?			R*	L01
6.	Does the normalized difference show negative bias between -1.96 and -2.58?			J	L02
7.	Does the normalized difference show negative bias less than -2.58?			R*	L02
8.	List the calculated normalized difference: _____				

Action: If deficiencies are found, qualify all samples in the preparation batch as indicated above. Indicate which samples were affected below.

Sample	Frequency	Matrix	Positive Bias	Negative Bias

* Evaluate other QC prior to qualifying "R". See Attachment H for guidance on qualification for multiple QC deficiencies.

$LCS_{meas.}$ = Measured LCS results

$LCS_{exp.}$ = Expected result of LCS

$$\frac{LCS_{meas.} - LCS_{exp.}}{\sqrt{(TPU_{meas.})^2 + (TPU_{exp.})^2}}$$

$TPU_{meas.}$ = 1s Total Propagated Uncertainty of measured result

$TPU_{exp.}$ = 1s Total Propagated Uncertainty of expected result

6.0 Laboratory Duplicate

Validation Step		Y	N	Q	C
1.	Has at least one laboratory duplicate been prepared with up to 20 samples?			--	D02
2.	Is the normalized absolute difference between the sample and laboratory duplicate values greater than 1.96?			J	D01
3.	List the calculated normalized difference: _____				
Action:	If deficiencies are found, qualify the samples in the preparation batch as indicated above. Indicate which samples were affected below.				

Sample ID	Duplicate ID	Frequency (check)	Statistical test shows significant difference
_____	_____	___	___

Comments:_____

* Evaluate other QC prior to qualifying. See Attachment H for guidance on qualification for multiple QC deficiencies.

S = Sample result

D = Laboratory duplicate result

$$\frac{S - D}{\sqrt{(TPU_S)^2 + (TPU_D)^2}}$$

TPU_s = 1s Total Propagated Uncertainty of the sample

TPU_D = 1s Total Propagated Uncertainty of the duplicate

7.0 Matrix Spike (MS)

Validation Step		Y	N	Q	C
1.	Has at least one MS been prepared up to 20 samples?			J	M04
2.	Is the normalized difference between the measured and expected values of the MS within -1.96 and 1.96?			--	--
3.	Does the normalized absolute difference show positive bias between 1.96 and 2.58?			J	M01
4.	Does the normalized absolute difference show positive bias greater than 2.58?			R*	M01
5.	Does the normalized absolute difference show negative bias between -1.96 and -2.58?			J	M02
6.	Does the normalized absolute difference show negative bias less than -2.58?			R*	M02
7.	List the calculated normalized difference: _____				

Action: If deficiencies are found, qualify the samples in the preparation batch as indicated above. Indicate which samples were qualified below.

Sample ID	MS ID	Frequency	Positive Bias	Negative Bias
_____	_____	_____	_____	_____

Comments:_____

* Evaluate other QC prior to qualifying "R". See Attachment H for guidance on qualification for multiple QC deficiencies.

SSR = Spiked Sample Result

SR = Sample Result (unspiked)

ER = Expected Result

$$\frac{(SSR - SR) - ER}{(TPU_{SSR})^2 + (TPU_{SR})^2 + (TPU_{ER})}$$

TPU_{ssr} = 1s Total Propagated Uncertainty of Spiked Sample Result

TPU_{SR} = 1s Total Propagated Uncertainty of Sample Result

TPU_{ER} = 1s Total Propagated Uncertainty of Expected Result

8.0 Method Blank

Validation Step		Y	N	Q	C
1.	Has at least one method blank been prepared with up to 20 samples?			J	B05
2.	Is the method blank the same matrix as the samples in the reporting batch?			--	B02
3.	Is the normalized absolute difference of the method blank and sample greater than 2.58?			--	--
4.	Is the normalized absolute difference of the method blank and sample between 1.96 and 2.58?			J	B06
5.	Is the normalized absolute difference of the method blank between 0 and 1.96?			R*	B06

Action: If deficiencies are found, qualify the affected samples as indicated above. Indicate which samples were affected below.

Sample ID	Matrix	If statistical test shows no significant difference between blank and sample, list the calculated normalized difference for each affected sample.
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

* Evaluate other QC prior to qualifying "R". See Attachment H for guidance on qualification for multiple QC deficiencies.

S = Sample result

B = Method blank result

$$\frac{|S-B|}{\sqrt{(TPU_S)^2+(TPU_B)^2}}$$

TPU_S = 1s Total Propagated Uncertainty of Sample

TPU_B = 1s Total Propagated Uncertainty of Method Blank

9.0 Chemical Yield - Tracers and Carriers

Validation Step		Y	N	Q	C
1.	Is yield reported for all samples and QC samples in the reporting batch?				Y04
2	Are percent recovery criteria satisfied for all yield results?			J/R	Y01, Y02, Y03
Action:	If deficiencies are found, qualify the affected samples as indicated above. Indicate which samples were affected below.				

Sample	% Recovery
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

10.0 Validation of Sample Measurements

Validation Step		Y	N	Q	C
1.	Are sample results the MDC?			U	Q15
2.	Are sample results their 2s counting uncertainty?			U	Q16
3.	Do negative sample results have absolute values greater than their 2 counting uncertainty?			J	Q17
4.	For gas proportional systems, was the sample weight equal or less than the greatest weight indicated on the mass attenuation curve?			J	Q20
5.	Each sample-specific TPU is < 80% of its respective sample result.			J	Q23
Action: Indicate the samples requiring qualification below.					
Action: According to the amount of manual transcription done by the laboratory, recalculate a percentage of all sample results within the reporting batch, starting with 10% of sample results, increasing until no errors are discovered. Show calculations on a separate page.					
Deficiency Description					

11.0 Spectral Interpretation — Gamma Analyses

Validation Step	Y	N	Q	C
1. Do isotopes of the same radionuclide show secular equilibrium?				Q21
2. Are all detected peaks correctly identified?			R	Q01
3. Overlap of peaks is not significant.			J/R	Q02, Q18
Action: Indicate the samples requiring qualification below.				
Deficiency Description				

12.0 Spectral Interpretation — Alpha Analyses

Validation Step	Y	N	Q	C
1. Are target peaks within the energy range of interest?			R	Q22
2. Peak overlap through tailing does not preclude quantitation.			J/R	Q02, Q18

Action: Indicate the samples requiring qualification below.

Deficiency Description

Attachment D
DATA VALIDATION QUALIFIERS, REASON CODES, AND QUALIFICATION CODES

Radiological Analytical Data

Reason codes should be used with all qualifiers placed on analytical data.

U Analyte compound or nuclide considered not detected above the reported detection limit

J Analyte compound or nuclide identified; the associated numerical value is approximated

UJ Analyte, compound or nuclide not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.

R Result is not usable for its intended purpose

= "Equals" sign, indicates that no qualifier is necessary

Only radiochemical validation codes are presented in this appendix.

Method Blank

B01 Concentration of contaminant in the blank at a level = the qualification level.
B02 Method Blank was not the same matrix as the analytical samples.
B03 Gross contamination exists.
B05 Blanks were not analyzed at appropriate frequency.
B06 Sample not significantly different than radiochemical method blank.
B07 Blank data not reported.
B08 Other (describe in comments)

Calibration

C07 Calibration data not reported
C08 Calibration not performed.
C09 Chemical resolution criteria were not satisfied.
C10 Calibration standard matrix not the same as sample matrix
C27 Calibration verification efficiency outside control criteria
C28 Calibration verification background outside control criteria
C29 Calibration verification energy outside control criteria
C30 Calibration verification peak resolution outside control criteria
C31 Other (describe in comments)

Laboratory Duplicate

D01 Significant difference between sample and duplicate.
D02 Laboratory duplicate was not analyzed at the appropriate frequency
D04 Laboratory duplicate data not reported
D05 Other (describe in comments)

Evidentiary Concerns

E01 Custody of Sample in Question.
E02 Standard not Traceable.
E03 Other (describe in comments)

General

G01 Professional judgement was used to qualify the data.
G02 Other (describe in comments)

Holding Times

H03 Analysis holding times were exceeded.
H04 Analysis holding times were grossly exceeded.
H05 Samples were not preserved properly.
H07 Sample temperature exceeded criteria prior to preparation
H08 Other (describe in comments)

Laboratory Control Sample

L01 LCS recovery above upper control limit.
L02 LCS recovery below lower control limit.
L03 LCS was not analyzed at appropriate frequency.
L04 LCS not the same matrix as the analytical samples.
L05 LCS data not reported
L06 Other (describe in comments)

Matrix Spike and MS/MSD

M01 MS and/or MSD recovery above upper control limit.
M02 MS and/or MSD recovery below lower control limit.
M04 MS and/or MS/MSD not analyzed at the appropriate frequency
M05 MS and/or MS/MSD data not reported
M06 Other (describe in comments)

Instrument Performance

P01 High background levels or a shift in the energy calibration were observed
P02 Extraneous peaks were observed.
P03 Loss of resolution was observed.
P04 Peak-tailing or peak splitting that may result in inaccurate quantitation were observed
P07 Other (describe in comments)

Quantitation

Q01 Peak Misidentified
Q02 Target analyte affected by interfering peak.
Q03 Qualitative criteria were not satisfied.
Q04 Cross contamination occurred.
Q12 No raw data were provided to confirm quantitation.
Q13 MDC > RDL
Q14 Inappropriate aliquot sizes were used.
Q15 Sample result < MDC.
Q16 Sample result < 2s uncertainty.
Q17 Negative result.
Q18 Compounds were not adequately resolved.
Q19 Sample geometry different from calibration geometry.
Q20 Sample weight greater than greatest weight on mass attenuation curve.
Q21 Isotopes of same radionuclide do not show equilibrium
Q22 Peak not within appropriate energy range
Q23 Counting uncertainty = 80% of sample result
Q24 Raw data anomaly
Q25 Other (describe in comments)

Radiochemical Yield

Y01 Radiochemical tracer yield was above the upper control limit
Y02 Radiochemical tracer yield was below the lower control limit
Y03 Radiochemical tracer yield was zero.
Y04 Radiochemical yield data was not present.
Y05 Other (describe in comments)

Attachment E
RADIOCHEMICAL DATA VALIDATION REQUIREMENTS

Purpose: This worksheet is the mechanism to be used to identify and document the implementation of procedure **PROCEDURE NAME.**

Instructions: One worksheet must be completed for each fraction and analytical method (e.g. alpha spectrometry, gross alpha and beta). The completed worksheet shall be forwarded to the ER Data Quality Program for approval. Full-size forms may be obtained from the ER Data Quality Programs Reference Library (423/241-4677).

If the requirements of this procedure, are adopted as-is, without the necessity of approved alternatives, enter "as-is for review item 1 in the implementation column and complete the project sign-off section at the bottom of the form. No other entries are needed.

If project-specific implementations are necessary,

1) Identify review items that will be directly implemented and enter as-is in the respective rows of the Implementation column.
2) Identify review items that will not be considered by verifiers or validators during the implementation of this procedure, and enter n/a in the respective rows of the Implementation column and reference attached materials documenting the reason for the exclusion of these review items in the Comment column.
3) Identify review items that will be implemented with project-specified alternative actions and enter Alternative in the respective rows of the Implementation column. In the Comment column, reference attached materials that document the need for the alternative actions and specify the actions to be implemented upon approval. This alternative description should cover the following: Deliverables, Frequency, Performance Criteria, Verification step(s), Validation step(s).
4) Complete the project sign-off section at the bottom of the worksheet and forward to the ER Data Quality Program for approval.

Fraction and Method:_____

	Implement	Comment
1. All requirements of ES-A-2209, Rev. 0 will be implemented:	____	_____
2. Holding time	____	_____
3. Initial calibration Gas Proportional Counting	____	_____
Liquid Scintillation Counting	____	_____
Alpha Spectrometry	____	_____
Gamma Spectrometry	____	_____
4. Calibration verification Gas Proportional Counting	____	_____
Liquid Scintillation Counting	____	_____
Alpha Spectrometry	____	_____
Gamma Spectrometry	____	_____
5. Standard Traceability	____	_____
6. Laboratory Control Sample	____	_____
7. Laboratory Duplicate	____	_____

8. Matrix Spike ____ – _____

9. Method Blank ____ – _____

10. Chemical Yield ____ – _____

11. Required Detection Limits ____ – _____

12. Nuclide Identification and Quantitation ____ – _____

13. Spectral Interpretation
    Alpha Spectrometry ____ – _____

    Gamma Spectrometry ____ – _____

The _____ project will approach radiochemical data validation with a strategy consistent with ES-A-2209, Rev. 0, and/or with specific alternative(s) described on the attached pages.

_____ _____

Signature Date

Attachment F
RECOMMENDED SURVEILLANCE CONSIDERATIONS FOR GAMMA ALGORITHMS

Radionuclide identification algorithms used by the laboratory should be evaluated during laboratory audits. Consideration should be given to the following:

1. The width of the energy-search windows for radionuclide identification to ensure a minimum of peak misidentifications,

2. The criteria by which the laboratory evaluates sample-specific peak resolution,

3. Documentation of the source of the isotope identification library used by the source [Most software packages come with a master library which users can pare down to one more useful in day to day analyses.],

4. Training records of those persons(s) using the peak identification respective software,

5. SOP documentation of the parameters used by the software in the analysis of gamma spectra,

6. Existence of a SOP documentation for the internal review of the reported data,

7. If the software package is capable of using pre-programmed macros to automate the spectral analysis, evaluate the existence of SOP documentation of these macros,

8. If the software package uses various flags to denote peak quality or analysis results, the analyst using the software should be able to explain the meaning of the flags and their impact on data quality.

9. Existence of a SOP to independently (within-lab) verify the gamma spectral library.

Attachment G
RADIOCHEMICAL EQUATIONS

Pages 57-62 Provide Equations For Method Blank Corrected Sample Concentrations
Pages 64-68 Provide Equations For Sample Concentrations Without Blank Subtraction

The equations in this appendix are provided as examples.
It is likely that laboratory data will be reported with variations of these equations.
These calculations should be regarded as guidance only.

Sample Activity Concentration — (Method Blank Corrected Sample Concentrations)

$$ACT_S = \frac{NCR_S}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

ACT_S	=	Sample Activity Concentration (pCi/g or pCi/L)
NCR_S	=	Net Sample Count Rate in cpm
2.22	=	Factor for Converting dpm to pCi
EFF	=	Detector Efficiency (Fraction)
ALI	=	Sample Aliquot Volume or Mass (g or L)
ABN_S	=	Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
R	=	Sample Tracer/Carrier (Chemical) Recovery
?	=	Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute ?]
t	=	Time from Sample Collection to Radionuclide Separation or Mid-point of Count Time (Same units as half-life)
CF	=	Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Net Sample Count Rate (NCR) and s_{NCR}— (<u>Method Blank Corrected</u> Sample Concentrations)

$$NCR = \left(\frac{C_{GS}}{T_{GS}} - \frac{C_{SB}}{T_{SB}}\right) - \left[\left(\frac{C_{GB}}{T_{GB}} - \frac{C_{BB}}{T_{BB}}\right) * \left(\frac{EFF_{SD}}{EFF_{BD}}\right) * \left(\frac{R_S}{R_B}\right)\right]$$

Where:

C_{GS}	=	Sample Counts
T_{GS}	=	Sample CountTime (minutes)
C_{SB}	=	Background Counts
T_{SB}	=	Background CountTime (minutes)
C_{GB}	=	Gross Method Blank Counts
T_{GB}	=	Gross Method Blank CountTime (minutes)
C_{BB}	=	Method Blank Background Counts
T_{BB}	=	Method Blank Background CountTime (minutes)
EFF_{SD}	=	Efficiency of the Sample Detector
EFF_{BD}	=	Efficiency of the Method Blank Detector
R_S	=	Sample Tracer/Carrier Recovery Fraction
R_B	=	Method Blank Tracer/Carrier Recovery Fraction

Uncertainty of Net Count Rate

$$\sigma^2_{NCR} = \left[\frac{C_{GS}}{T^2_{GS}} + \frac{C_{SB}}{T^2_{SB}}\right] + \left\{\left[\frac{C_{GB}}{T^2_{GB}} + \frac{C_{BB}}{T^2_{BB}}\right] * \left(\frac{EFF_{SD}}{EFF_{BD}}\right)^2 * \left(\frac{R_S}{R_B}\right)^2\right\}$$

$$+ \left\{\left[\left(\frac{C_{GB}}{T_{GB}} - \frac{C_{BB}}{T_{BB}}\right) * \left(\frac{EFF_{SD}}{EFF_{BD}}\right) * \left(\frac{R_S}{R_B}\right)\right]^2 * \left[\left(\frac{\sigma_{EFF_{SD}}}{EFF_{SD}}\right)^2 + \left(\frac{\sigma_{EFF_{BD}}}{EFF_{BD}}\right)^2 + \left(\frac{\sigma_{R_S}}{R_S}\right)^2 + \left(\frac{\sigma_{R_B}}{R_B}\right)^2\right]\right\}$$

$$\sigma_{NCR} = \sqrt{\sigma^2_{NCR}}$$

Calculation of Recovery - Radiometric— (Method Blank Corrected Sample Concentrations)

$$R = \frac{\left(\frac{C_{GT}}{T_{GT}} - \frac{C_{TB}}{T_{TB}}\right)}{EFF * ABN_T * AMT_T} = \frac{NCR_T}{EFF * ABN_T * AMT_T}$$

Where:

R	=	Tracer Recovery
C_{GT}	=	Gross Count of Tracer
T_{GT}	=	Tracer CountTime (minutes)
C_{TB}	=	Background Count of Tracer [Region of Interest (ROI)]
T_{TB}	=	Background CountTime (minutes)
EFF	=	Detector Efficiency Fraction
AMT_T	=	Amount of Tracer Activity Added (dpm)
NCR_T	=	Net Count Rate of Tracer (cpm)
ABN_T	=	Abundance Fraction of the Tracer Emissions used for Quantification of the Tracer

Note: It is assumed that the tracer half-life is long enough to be an insignificant uncertainty contributor. If the tracer has a relatively short half-life, then it must be considered and these equations modified. Likewise, uncertainty in the time is also considered to be an insignificant contributor.

Calculation of RE_R - Radiometric— (Method Blank Corrected Sample Concentrations)

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 + \left(\frac{\sigma_{EFF}}{EFF}\right)^2 + \left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2$$

$$\left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 = \frac{\left(\frac{C_{GT}}{T_{GT}^2} + \frac{C_{TB}}{T_{TB}^2}\right)}{NCR_T^2}$$

$$\left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2 = \left(\frac{\sigma_{STS_T}}{STS_T}\right)^2 + \left(\frac{\sigma_{MASS_T}}{MASS_T}\right)^2 + \left(\frac{\sigma_{VOL_T}}{VOL_T}\right)^2 + \Sigma\left(\frac{\sigma_{DIL_T}}{DIL_T}\right)^2 + \Sigma\left(\frac{\sigma_{ALI_T}}{ALI_T}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

RE_R	=	Relative Error of the Tracer Recovery
s_R	=	Standard Deviation of the Tracer Recovery
s_{NCR_T}	=	Standard Deviation of the Tracer's Net Count Rate
s_{EFF}	=	Standard Deviation of the Detector Efficiency
s_{AMT_T}	=	Standard Deviation of the Amount of Tracer Activity Added
s_{STS_T}	=	Standard Deviation of the Amount of Tracer Activity Taken for Stock Tracer Solution (provided with certificates received with standards)
s_{MASS_T}	=	Standard Deviation of the Mass of Standard Solution Used to Prepare Stock Tracer Solution
s_{DIL_T}	=	Standard Deviation of the Volume(s) of the Dilution(s) Made to Prepare the Working Tracer Solution
s_{VOL_T}	=	Standard Deviation of the Volume of the Stock Tracer Solution
s_{ALI_T}	=	Standard Deviation(s) of the Aliquot(s) of Tracer Solution(s) Diluted to Prepare Working Tracer Solution
C_{GT}	=	Gross Count of Tracer
T_{GT}^2	=	Square of Tracer CountTime (minutes)
C_{TB}	=	Background Count of Tracer [Area or Region of Interest (ROI)]
T_{TB}^2	=	Square of Background CountTime (minutes)
R	=	Tracer Recovery
NCR_T	=	Net Count Rate of the Tracer
EFF	=	Detector Efficiency
AMT_T	=	Amount of Tracer Activity Added (dpm)
STS_T	=	Amount of Tracer Activity (dpm) in Stock Tracer Solution
$MASS_T$	=	Mass (grams) of Standard Solution Used to Prepare Stock Tracer Solution
VOL_T	=	Volume of Tracer Solution Added
DIL_T	=	Volume(s) of Dilution(s) Made to Prepare the Working Tracer Solution
ALI_T	=	Aliquot(s) of Tracer Solution(s) Taken to Prepare Serial Tracer Solution Dilution(s)

Note: Certificates, such as those from NIST, may give two or even three sigma uncertainty. Only one sigma should be used for s_{STS_T}.

Calculation of Recovery and RE_R - Gravimetric — (Method Blank Corrected Sample Concentrations)

$$R = \frac{WT_c}{CONC_{CS} * VOL_{CS}}$$

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{PPT.WT.}}{PPT.WT.}\right)^2 + \left(\frac{\sigma_{CONC_{CS}}}{CONC_{CS}}\right)^2 + \left(\frac{\sigma_{VOL_{CS}}}{VOL_{CS}}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

R	=	Carrier Recovery
RE_R	=	Relative Error in Recovery
WT_c	=	Weight of Carrier Present in Final Precipitate
$CONC_{cs}$	=	Concentration of Carrier Solution
VOL_{cs}	=	Volume of Carrier Solution Added
$s_{PPT.WT.}$	=	Standard Deviation in Weight of Precipitate
s_{CONC}	=	Standard Deviation in Carrier Concentration
s_{VOL}	=	Standard Deviation in Carrier Volume
PPT. WT.	=	Weight of Final Carrier Precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

$$CU_{1.96\sigma} = \frac{1.96 * (\sigma_{NCR_S})}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

s_{NCR_S}	=	Standard Deviation of the Net Sample Count Rate
EFF	=	Detector Efficiency
ALI	=	Sample Aliquot Volume or Mass
R	=	Sample Tracer/Carrier Recovery
ABN_S	=	Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
?	=	Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute ?]
t	=	Time from Sample Collection To Radionuclide Separation or Mid-point of CountTime (Same units as half-life)
CF	=	Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Sample Activity Concentration Total Propagated Uncertainty (TPU)—(<u>Method Blank Corrected</u> Sample Concentrations)

$$TPU_{1\sigma} = \sigma_{ACT} = \frac{\sqrt{\sigma_{NCR_S}{}^2 + (NCR)^2 * \left(RE^2_{EFF} + RE^2_{ALI} + RE^2_R + \Sigma\, RE^2_{CF}\right)}}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

EFF	=	Detector Efficiency
ALI	=	Sample Aliquot Volume or Mass
R	=	Sample Tracer/Carrier Recovery
ABN_S	=	Abundance Fraction of the Emissions Used for Identification/Quantification
$s^2_{NCR_S}$	=	Variance of the Net Sample Count Rate
NCR	=	Net Sample Count Rate
RE^2_{EFF}	=	Square of the Relative Error of the Efficiency Term
RE^2_{ALI}	=	Square of the Relative Error of the Aliquot
RE^2_R	=	Square of the Relative Error of the Sample Recovery
RE^2_{CF}	=	Square of the Relative Error of Other Correction Factors
?	=	Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute ?]
t	=	Time from Sample Collection to Radionuclide Separation or Mid-Point of Count Time (Same units as half-life)
CF	=	Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Sample Activity Concentration — Sample Concentrations <u>Without Blank Subtraction</u>

$$ACT_B = \frac{NBCR_S}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

ACT_B = Sample Activity Concentration Without Method Blank Subtraction
$NBCR_S$= Net Sample Background-Corrected Count Rate
2.22 = Factor for Converting dpm to pCi
EFF = Detector Efficiency
ALI = Sample Aliquot Volume or Mass
ABN_S = Abundance Fraction of the Emissions Used for Identification/Quantification
R = Sample Tracer/Carrier Recovery
? = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute ?]
t = Time from Sample Collection to Radionuclide Separation or Mid-point of Count Time (Same units as half-life)
CF = Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Net Sample Count Rate ($NBCR_S$) and s_{NBCR_S} — Sample Concentrations <u>Without Blank Subtraction</u>

$$NBCR_S = \left(\frac{C_{GS}}{T_{GS}} \right)$$

Where:

$NBCR_S$ = Net Background Corrected Count Rate
C_{GS} = Sample Counts
T_{GS} = Sample Count Time (minutes)
C_{SB} = Background Counts
T_{SB} = Background Count Time (minutes)

s_{NBCR_S}

$$\sigma_{NBCR_S} = \left[\frac{C_{GS}}{T_{GS}^{2}} \right]^{\frac{1}{2}}$$

Calculation of Minimum Detectable Concentration (MDC) - general formula

$$MDC = \frac{4.65 * \sqrt{b/T}}{K} + \frac{2.71}{K * T}$$

Where:

b = background count rate
T = Sample Count Time (minutes)
K = instrument-specific and sample-specific correction factors (e.g. ALI * $e^{-?t}$ * R * EFF_S * ABN_S)

In using the above equation, the background and sample count times are either equivalent, or the background count time is greater than sample count time

Calculation of Recovery and RE_R - Radiometric — Sample Concentrations <u>Without Blank Subtraction</u>

$$R = \frac{\left(\frac{C_{GT}}{T_{GT}} - \frac{C_{TB}}{T_{TB}}\right)}{EFF * ABN_T * AMT_T} = \frac{NCR_T}{EFF * ABN_T * AMT_T}$$

Where:

R = Tracer Recovery
C_{GT} = Gross Count of Tracer
T_{GT} = Tracer CountTime (minutes)
C_{TB} = Background Count of Tracer Region of Interest (ROI)
T_{TB} = Background CountTime (minutes)
EFF = Detector Efficiency
AMT_T = Amount of Tracer Activity Added (dpm)
NCR_T = Net Count Rate of Tracer (cpm)
ABN_T = Abundance Fraction of the Tracer Emissions used for Quantification of the Tracer

RE_R - Radiometric — Sample Concentrations <u>Without Blank Subtraction</u>

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 + \left(\frac{\sigma_{EFF}}{EFF}\right)^2 + \left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2$$

$$\left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 = \frac{\left(\frac{C_{GT}}{T_{GT}^2} + \frac{C_{TB}}{T_{TB}^2}\right)}{NCR_T^2}$$

$$\left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2 = \left(\frac{\sigma_{STS_T}}{STS_T}\right)^2 + \left(\frac{\sigma_{MASS_T}}{MASS_T}\right)^2 + \left(\frac{\sigma_{VOL_T}}{VOL_T}\right)^2 + \Sigma\left(\frac{\sigma_{DIL_T}}{DIL_T}\right)^2 + \Sigma\left(\frac{\sigma_{ALI_T}}{ALI_T}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

RE_R = Relative Error of the Tracer Recovery
s_R = Standard Deviation of the Tracer Recovery
s_{NCR_T} = Standard Deviation of the Tracer's Net Count Rate
s_{EFF} = Standard Deviation of the Detector Efficiency
s_{AMT_T} = Standard Deviation of the Amount of Tracer Activity Added
s_{STS_T} = Standard Deviation of the Amount of Tracer Activity Taken for Stock Tracer Solution (provided with certificates received with standards)
s_{MASS_T} = Standard Deviation of the Mass of Standard Solution Used to Prepare Stock Tracer Solution
s_{DIL_T} = Standard Deviation of the Volume(s) of the Dilution(s) Made to Prepare the Working Tracer Solution
s_{VOL_T} = Standard Deviation of the Volume of the Stock Tracer Solution
s_{ALI_T} = Standard Deviation(s) of the Aliquot(s) of Tracer Solution(s) Diluted to Prepare Tracer Working Solution
C_{GT} = Gross Count of Tracer
T_{GT}^2 = Square of Tracer Count Time
C_{TB} = Background Count of Tracer ROI
T_{TB}^2 = Square of Background Count Time
R = Tracer Recovery
NCR_T = Net Count Rate of the Tracer
EFF = Detector Efficiency
AMT_T = Amount of Tracer Activity Added (dpm)
STS_T = Amount of Tracer Activity (dpm) in Stock Tracer Solution
$MASS_T$ = Mass (grams) of Standard Solution Used to Prepare Stock Tracer Solution
VOL_T = Volume of Tracer Solution Added
DIL_T = Volume(s) of Dilution(s) Made to Prepare the Working Tracer Solution
ALI_T = Aliquot(s) of Tracer Solution(s) Taken to Prepare Serial Tracer Solution Dilution(s)

Note: Certificates, such as those from NIST, may give two or even three sigma uncertainty. Only one sigma should be used for s_{STS_T}.

Calculation of Recovery and RE_R - Gravimetric — Sample Concentrations <u>Without Blank Subtraction</u>

$$R = \frac{WT_c}{CONC_{CS} * VOL_{CS}}$$

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{PPT.WT.}}{PPT.WT.}\right)^2 + \left(\frac{\sigma_{CONC_{CS}}}{CONC_{CS}}\right)^2 + \left(\frac{\sigma_{VOL_{CS}}}{VOL_{CS}}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

R	=	Carrier Recovery
RE_R	=	Relative Error in Recovery
WT_C	=	Weight of Carrier Present in Final Precipitate
$CONC_{CS}$	=	Concentration of Carrier Solution
VOL_{CS}	=	Volume of Carrier Solution Added
$s_{PPT.WT.}$	=	Standard Deviation in Weight of Precipitate
s_{CONC}	=	Standard Deviation in Carrier Concentration
s_{VOL}	=	Standard Deviation in Carrier Volume
PPT. WT.	=	Weight of Final Carrier Precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

$$CU_{B1.96\sigma} = \frac{1.96 * (\sigma_{NBCRs})}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

s_{NBCR_S}	=	Standard Deviation of the Net Background Corrected Count Rate
EFF	=	Detector Efficiency
ALI	=	Sample Aliquot Volume or Mass
R	=	Sample Tracer/Carrier Recovery
ABN_S	=	Abundance Fraction of the Emissions Used for Identification/Quantification
?	=	Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute ?]
t	=	Time from Sample Collection To Radionuclide Separation or Mid-point of Count Time (Same units as half-life)
CF	=	Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Sample Activity Concentration Total Propagated Uncertainty (TPU)— Sample Concentrations <u>Without Blank Subtraction</u>

$$TPU_{B1\sigma} = \sigma_{ACT} = \frac{\sqrt{\sigma_{NBCR_S}{}^2 + (NBCR_S)^2 * \left(RE^2_{EFF} + RE^2_{ALI} + RE^2_R + \Sigma\, RE^2_{CF}\right)}}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

EFF	=	Detector Efficiency
ALI	=	Sample Aliquot Volume or Mass
R	=	Sample Tracer/Carrier Recovery
ABN_S	=	Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
$s^2_{NBCR_S}$	=	Variance of the Net Background Corrected Count Rate
$NBCR_S$	=	Net Background Corrected Count Rate
RE^2_{EFF}	=	Square of the Relative Error of the Efficiency Term
RE^2_{ALI}	=	Square of the Relative Error of the Aliquot
RE^2_R	=	Square of the Relative Error of the Sample Recovery
RE^2_{CF}	=	Square of the Relative Error of Other Correction Factors
?	=	Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute ?]
t	=	Time from Sample Collection to Radionuclide Separation or Mid-Point of Count Time (Same units as half-life)
CF	=	Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Note: For methods using a tracer or carrier, the inclusion of efficiency and recovery terms in the equation above may result in overestimation of the TPU.

Attachment H
MAPPING FOR MULTIPLE QUALITY DEFICIENCIES

These tables represent validation qualification decisions made based on radioanalytical data quality considerations only. Data quality needs (e.g. risk assessment, remediation technologies) must be considered when using the guidance in these tables. For example, quantitative data needs may necessitate that data be rejected due to multiple quality deficiencies, but qualititative data needs may indicate that the same data should only be qualified estimated.

Table H.1 provides a mapping scenario for qualification guidance. For example, if the Laboratory Control Sample shows a high bias, and chemical yield is also high, choose the letters "A" and "E" and reference Table H.2 for guidance on qualification based on those quality deficiencies. The differing separation lines in table H.1 indicate relationships among the quality indicators. Double lines indicate an "and" function, in that any combination is possible. Thick lines indicate that a single QC sample can only indicate a bias in one direction.

"High" and "low" bias in table H.1 represent the confidence level by which the decision has been made to qualify affected data (Section F).

Table H.1

Lab Control Sample		Matrix Spike		Chemical Yield		Method Blank Contamination	Lab Duplicate Poor Precision
High Bias	Low Bias	High Bias	Low Bias	High	Low		
A	B	C	D	E	F	G	H

Table H.2
Qualification Guidance for 2 Simultaneous Quality Deficiencies

Combination	Qualification	
	<MDC	=MDC
AC	None	R
AD	UJ	J
AE	None	R
AF	UJ	J
AG	None	J
AH	None	J
BC	UJ	J
BD	R	J
BE	UJ	J
BF	R	J
BG	UJ	J
BH	UJ	J
CE	None	R
CF	UJ	J
CG	None	R
CH	None	R
DE	UJ	J
DF	R	J
DG	UJ	R
DH	UJ	R
EG	None	R
EH	None	J
FG	UJ	J
FH	UJ	J
GH	UJ	J

Table H.3
Qualification Guidance for 3 Simultaneous Quality Deficiencies

Combination	Qualification	
	<MDC	=MDC
ACE	None	R
ACF	UJ	R
ACG	None	R
ACH	None	R
ADE	UJ	R
ADF	R	R
ADG	UJ	R
ADH	UJ	R
AEG	None	R
AEH	None	R
AFG	UJ	R
AFH	None	R
BCE	UJ	R
BCF	UJ	R
BCG	UJ	R
BCH	UJ	R
BDE	R	R
BDF	R	R
BDG	R	R
BDH	R	R
BEG	UJ	R
BEH	UJ	R
BFG	R	R
BFH	R	R
CEG	None	R
CEH	None	R
CFG	UJ	R
CFH	UJ	R
DEG	UJ	R
DEH	UJ	R
DFG	R	R
DFH	R	R
EGH	None	R
FGH	UJ	R

Table H.4
Qualification Guidance for 4 Simultaneous Quality Deficiencies

Combination	Qualification	
	<MDC	=MDC
ACEG	None	R
ACFG	UJ	R
ACEH	None	R
ACFH	UJ	R
ADEG	UJ	R
ADFG	R	R
ADEH	UJ	R
ADFH	R	R
BCEG	UJ	R
BCFG	R	R
BCEH	UJ	R
BCFH	R	R
BDEG	R	R
BDFG	R	R
BDEH	R	R
BDFH	R	R
CEGH	None	R
CFGH	UJ	R
DEGH	UJ	R
DFGH	R	R

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED:

Paducah Site-Wide
Procedure Type
(Company, Functional, Project, Subcontractor)

PA-5105: Volatile and Semivolatile Data Verification & Validation
(Procedure Number and Title)

YY 4-13-06
~~2/2/1999~~ 4-13-1999
(Procedure Date)

Rev. 0
(Rev. Number)

Reason for Changes: Transitioning BJC Procedures

SECTION	DESCRIPTION OF CHANGE
General	When reference is made to Lockheed Environmental Management and/or Bechtel Jacobs LLC personnel or contract, change the reference to Paducah Remediation Services (LLC) personnel or contract. Note that the procedure numbers will not change until after the first revision by PRS LLC.
General	Change Bechtel Jacobs LLC Environmental Management (EM) Projects to Paducah Remediation Services (LLC) Environmental Management (EM) Projects.
General	Attached is a cross walk from the Lockheed Environmental Management and the old Bechtel Jacobs LLC procedure numbers to the current Bechtel Jacobs LLC procedures. The procedure will be re-numbered with it is revised after transition.
Purpose	Delete the following sentence "This procedure applies to all volatile and semivolatile data verification and validation activities performed by or for the ER Program Organization at the East Tennessee Technology Park (ETTP), Oak Ridge National Laboratory (ORNL), Y-12 Plant (Y-12), Paducah Gaseous Diffusion Plant (PGDP), and Portsmouth Gaseous Diffusion Plant (PORTS) sites, as well as those performed by or for ER's Off-Site Program." and replace it with "This procedure applies to all volatile and semivolatile data verification and validation activities performed by or for the ER Program Organization Paducah Gaseous Diffusion Plant (PGDP)."

Source Documents	This procedure was last revised in 1999 and the source data lists Martin Marietta Energy Systems Inc. procedures. It is anticipated that PRS LLC will review and up date this procedure, and at that time update the references to match the existing work flow.

Initiator/Originator: Darryl Poteet / [signature] — 4/7/06
(Print Name / Sign Name) — Date

REVIEWED BY: [signature] — 4/7/06
Discipline/Task Manager — Date

APPROVED BY: Diane M. Daniel — 4/7/06
Project Manager — Date

BJC to PRS Position/Title/Organization Matrix
for PA-5105 Volitile and Semivolitile Data Verification and Validation

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
Project Manager	→	Project Manager
Data Verifier	→	Data Entry Specialist
Data Validator	→	Statistician/Validator
Data Validation Coordinator	→	Statistician/Validator
ER-DQP Data Validation Coordinator	→	Environmental Monitoring/Sample Manager
BJC Watershed Representative	→	Sr. Scientist
	→	
Data Management Coordinator		Data Manager/Network Specialist
Record Management Coordinator	→	Data Entry Specialist
	→	
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I. PURPOSE/SCOPE

This procedure defines the minimum requirements, responsibilities and methodology for the volatile and semivolatile data verification and validation processes within the Bechtel Jacobs Company Environmental Restoration (ER) Program Organization. This procedure applies to all volatile and semivolatile data verification and validation activities performed by or for the ER Program Organization at the East Tennessee Technology Park (ETTP), Oak Ridge National Laboratory (ORNL), Y-12 Plant (Y-12), Paducah Gaseous Diffusion Plant (PGDP), and Portsmouth Gaseous Diffusion Plant (PORTS) sites, as well as those performed by or for ER's Off-Site Program.

This procedure provides requirements for developing and implementing a validation methodology for volatile and semivolatile Contract Laboratory Program (CLP) and SW-846 (8260 and 8270) analytical methods primarily for analytes in aqueous and soil/sediment matrices. It is flexible enough to allow evaluation of data useability for project-specific Data Quality Objectives (DQOs). Data produced by analytical methods for which this procedure provides limited guidance (i.e., Appendix A of 40 CFR Part 136, *Protection of Environment, Appendix A - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, or "Superfund Low Concentration Statement of Work" methods) may necessitate development of modified criteria from this procedure; however, the general validation strategy outlined in this document should be applicable.

Specifications in this procedure should be incorporated into project documentation such as the Quality Assurance Project Plan, into contractual Statements of Work (SOW) between the project and the analytical laboratories, and into contractual validation SOWs between the project and the firm chosen to validate data. If validation is performed by individuals within the project, the SOW is not required but a mechanism to specify validation requirements is recommended. This procedure shall be used as a baseline to create project-specific checklists needed to perform volatile and semivolatile data verification and validation. Guidance in developing and implementing a validation plan is provided in the ER procedure ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects". Examples of checklists for implementing this procedure are provided in Appendices A and B.

In this procedure, the words "shall" and "must" are used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission, neither a requirement nor a recommendation. In conformance to this procedure, all verification and validation shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

II. REFERENCES

A. Source Documents

Data Management Plan for the Environmental Restoration Program, ES/ER/TM-88.

"Data Validation Plans for Environmental Restoration Projects," ERWM/ER-P2213, Bechtel Jacobs Company, LLC, Oak Ridge, Tennessee.

B. Other Documents

Data Quality Objectives Process for Superfund, EPA/540/G-93/071, September 1993.

Guidance for the Data Quality Objectives Process, EPA QA/G-4. August 1994

USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, EPA-540/R-94/012, February 1994.

USEPA Contract Laboratory Program Statement of Work for Organics Analysis, March 1990. Revision OLM03.1.

USEPA Test Methods for Evaluating Solid Waste, SW-846 third edition, November 1986. Revisions through Final Update III.

III. DEFINITIONS

Qualifier definitions and reason codes are listed in Appendix C.

Affected Sample Result: An affected sample result is a sample result considered to be affected significantly by a quality deficiency and is qualified accordingly through analytical data validation.

Analytical Data Validation: Analytical data validation is a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Analytical Data Verification: Analytical data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract which is performed by either the data generator or by an entity external to the data generator.

Batch: A batch is a group of samples prepared at the same time in the same location using the same method, not to exceed 20 samples of similar matrix

Bromofluorobenzene (BFB) : instrument performance check compound for volatile organics analysis by GC/MS

Calibration Check Compounds (CCC): CCC's are compounds used to verify the continued calibration of an instrument for the SW-846 analytical methodologies applied to volatile organics analysis (VOA) and semivolatile organics analysis (SVOA).

Case: A finite, usually predetermined number of samples collected over a given time period from a particular site. A case consists of one or more sample delivery groups (SDGs).

Chain of Custody (COC): The COC is the history of the transfer of samples from the time of sample acquisition through archival and disposal of samples.

Contract Required Quantitation Limit (CRQL): The CRQL is the minimum level of detection acceptable under the current contract.

Correctable Problem: Correctable problems are deficiencies within data packages which may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation.

- correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory.

- correctable problems revealed during validation are those deficiencies with analyses that can be solved by either a second preparation and/or analysis of a sample.

Decafluorotriphenylphosphine (DFTPP): DFTPP is used as the instrument performance check compound for semivolatile organics analysis by GC/MS.

Initial Calibration: Initial calibration, as described in this procedure, is defined as the standardization of a GC/MS instrument against a traceable standard of known identity and quantity. This standardization prevails until such time as analytical conditions are deemed out of acceptable control limits.

Internal Standard: Internal standards are non-target compounds added to every VOA and SVOA standard, blank, matrix spike, duplicate, and sample extract at a known concentration, prior to instrumental analysis. Internal standards are used as the basis for quantitation of the target compounds.

Laboratory Duplicate: The laboratory duplicate is a randomly chosen split of an field sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

Matrix Spike: The matrix spike is a split of a field sample in which one half of the split is spiked with a known amount of analyte of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation.

Method Blank: The method blank is a laboratory-generated sample of the same matrix as the analytical samples, but in absence of the analyte of interest. The purpose of a method blank is to monitor the presence of contamination of the analyte of interest in the sample preparation and analysis processes.

Non-Correctable Problem: Non-correctable problems are deficiencies within data packages which preclude the evaluation of data quality by predefined criteria. Non-correctable problems may be revealed during both data verification and data validation.

Quality-indicator Sample: Quality-indicator samples are those samples made ready in the laboratory which provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

Relative Percent Difference (RPD): RPD is the measure of precision between two values, defined as the absolute value of the difference between two values divided by the mean of the two values.

Relative Response Factor (RRF): RRF represents the response of a compound to an analytical instrument relative to the response of an associated standard.

Relative Standard Deviation (RSD): RSD is the measure of precision between multiple values, defined as the standard deviation of multiple values divided by the mean of the values.

Required Detection Limit (RDL): The RDL is a contractually-specified detection limit which, under typical analytical circumstances, should be achievable.

Sample Delivery Group (SDG): An SDG is defined by one of the following, whichever occurs first:

- Case of field samples
- Each 20 field samples within a case
- Each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the SDG.

Sample Result: A sample result, as described in this procedure, is a numeric denotation of the concentration, amount or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

Sample Quantitation Limit (SQL): SQLs are detection limits based on CRQLs, which have been modified due to deviations from analytical method specifications such as sample weight and extract volume or due to dilution or percent moisture.

Semivolatile Organics Analysis (SVOA) - Compounds analyzed by semivolatile analytical methods. These compounds are commonly referred to as Bases, Neutrals and Acids (BNAs).

Standard Reference Material (SRM): A material or substance of which one or more properties are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Surrogate: For semivolatiles and volatiles, non-target standard compounds added to every blank, sample matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring percent recovery. SVOA surrogates are brominated, fluorinated, or isotopically labeled compounds similar to the compounds of interest in chemical composition. VOA surrogates are brominated or deuterated compounds. Surrogates are not expected to be detected in environmental media.

System Performance Check Compounds (SPCC): Compounds used to establish the calibration of an instrument for the SW-846 analytical methodologies applied to VOA and SVOA.

Technical Holding Time: For volatile and semivolatile compounds, the time, expressed in calendar days, from sample collection to sample extraction (or purge), or from sample extraction to sample analysis.

Tentatively Identified Compounds (TIC)- TICs are compounds detected in samples that are not target compounds, internal standards, system monitoring compounds, or surrogates. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

Traceable Reference Material (TRM): A TRM is a NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).

Turn-around Time: Turn-around time is contractually-specified as the length of time which elapses between laboratory receipt of the field samples and subsequent data receipt by the client.

Validation Statement of Work: The validation SOW is a document prepared by the ER project to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

Volatile Organics Analysis (VOA)- method based on the purge and trap technique for organic compound analysis.

Well Characterized Reference Material (WCRM): The WCRM may be derived from a field sample which has been well characterized through multiple analyses, providing a high level of confidence of the concentration in the sample. The WCRM may be submitted to NIST for characterization and classification as a TRM.

IV. GENERAL INFORMATION

A. Data Verification

Data verification is defined as a systematic process, performed by either the data generator (on-site or fixed base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

If verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

Data verification, at the project level, may be conducted either by the data verifier or by the data validator, whichever expedites the review process. If verification is conducted independently of the validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and statement of work, in terms of the required deliverables. The second activity entails various checks of the quality of the data, the purpose of which is to alert the validator that a non-correctable problem has occurred and that data may need qualification. If verification is conducted by the validator, the first activity is conducted similarly, but the second activity may result in prompt qualification of data.

Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an after-the-fact identification of non-correctable deficiencies.

A verification report is written by the data verifier and takes, as input, the steps in this procedure that are listed as "Verification." The data verifier does not qualify data if verification criteria are not met but indicates in the verification report the circumstances surrounding correctable and non-correctable problems in the data package which will be transferred to the validator for possible qualification. The verification report must be in a standard format, and must remain associated with the analytical data package. A verification report which includes all verification checks in this procedure is provided in Appendix A.

B. Analytical Data Validation

Analytical data validation, including field and laboratory data review, is defined as a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data which may result in qualification of the data. Data validation is not performed by the analytical laboratory. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent, and if present, the level of uncertainty associated with the measurement, and occurs prior to drawing a conclusion from a body of data. Analytical data validation includes a technical review of a laboratory data package covering the evaluation of quality control samples, the identification and quantitation of analytes, and the effect of deficiencies in quality on analytical sample data.

A validation report that includes the results of validation activities must be completed by the validator and takes, as input, the verification report and the steps in this procedure that are listed as "Validation." A validation report must be completed for every SDG that passes through validation. To expedite writing the validation report, comments must include explanations for qualification only if the reason codes do not

adequately describe justification for qualification. Comments on data qualification for which reason codes adequately describe qualification reason are not necessary. A validation report which includes all validation checks in this procedure is provided in Appendix B.

Where professional judgement is used, a second opinion on choice of qualification of data should be sought. Consensus on professional judgement should be documented in the validation report.

V. REQUIREMENTS

A. General Requirements

1. This procedure will become effective thirty (30) days following the approval date to allow for distribution and initiation of training.

2. The actions described in this document must serve as the baseline for incorporation into project verification/validation activities. Project-specific verification/validation procedures applying to volatile and semivolatile compounds and analytical methods not covered in this document must be submitted to the ER Data Quality Program for review and approval prior to use. Verification and validation activities subcontracted outside Bechtel Jacobs Company must adopt requirements consistent with those provided in this procedure.

B. Analytical Statement of Work

Analytical SOW Requirements:

Implementation of this procedure is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory Statement of Work (SOW). Deliverable requirements specified in the analytical SOW must be consistent with the requirements of this procedure, and must be in agreement with the Analytical Master Specifications of the BJC Terms and Conditions.

C. Validation Statement of Work Requirements

The validation SOW must be written consistent with the requirements and specifications of this procedure. The validation SOW is prepared by the ER project team and functions as the mechanism by which validation implementation requirements are communicated from the project team to the validation organization.

D. Validation Data Qualification Requirements

When qualification conditions are met, a qualifier and reason code(s) shall be physically and/or electronically associated with the affected result(s). If more than one reason code is applied, each should be separated by commas. The listing of available reason codes is presented in Appendix C. If data are not qualified, an equals sign ("=") is entered on the sample result. As directed in the validation SOW or as appropriate, reason codes may be applied without qualifiers.

E. Validation Strategy Worksheet Requirements

The worksheet presented in Appendix D must be completed by the project to identify the working volatile and semivolatile data validation approach. For worksheet items where direct implementation of this procedure is not appropriate, an alternative approach must be provided and must be submitted to the ER-DQP for approval. Establishment of the data validation strategy and plan is described in procedure ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects".

F. Validation Reporting Requirements

A verification report (Appendix A) and validation report (Appendix B) must be completed for every sample delivery group (SDG) that passes through validation. Both the verification and validation reports must be completed regardless of who performs the reviews.

VI. RESPONSIBILITIES

The responsibilities outlined in this section do not preclude an individual assuming multiple responsibilities.

A. ER-DQP Data Validation Coordinator

The ER-DQP Data Validation Coordinator is responsible for approval of alternative and non-routine verification and validation activities, and for providing training and technical support to ER projects in the areas of data verification and validation.

Responsibilities listed below denote activities at the project-level:

B. Project Manager

The Project Manager is responsible for oversight of the validation activities occurring within the project as a result of implementation of the requirements of this document.

C. Data Verifier

The Data Verifier is responsible for performing the verification actions listed in this procedure, including all items in Sect. VII listed as "Verification" and the writing of a verification report.

D. Data Validator

The Data Validator is responsible for performing the validation actions listed in this procedure, including all items in Sect. VII listed as "Validation" and the writing of the validation report.

E. Data Validation Coordinator

The Data Validation Coordinator is responsible for developing a data validation plan for the project, for incorporating the requirements of this procedure into the data validation plan for the project, for completing the validation implementation form (Appendix D), and for interfacing with the BJC Watershed Representative and laboratory concerning data package deficiencies.

F. BJC Watershed Representative

The BJC Watershed Representative is responsible for interfacing with the data validation coordinator to ensure that analytical statements of work incorporate deliverables required by this procedure so that data packages from the laboratory will be appropriate for verification and validation.

G. Records Management Coordinator

The Records Management Coordinator is responsible for interfacing with the data validation coordinator in transferring data packages and verification/validation reports, and in providing a system of storage and control of these documents.

H. Data Management Coordinator

The Data Management Coordinator is responsible for interfacing with the data validation coordinator in transferring validated sample data and in entering validated data into the project database.

VII. ACTION STEPS

The steps in this section define the processes of volatile and semivolatile data verification and validation.

Refer to Appendix C for qualifier and reason code descriptions. Refer to Appendix E for guidance on qualification due to multiple quality deficiencies. Refer to Appendix F for a listing of relevant equations to use with this procedure.

A. Overall Implementation of Validation

The following is a step-by-step approach to implement analytical data verification and validation activities for ER projects.

1. Validation Strategy and Plan Development

Data Validation Coordinator and Project Team

Develop validation strategy based on inputs identified through the DQO process according to ER procedure ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects".

Complete the Volatile and Semivolatile Data Validation implementation form (Appendix D).

Write a validation Statement of Work to communicate verification and validation requirements to the organization performing that work for the ER project.

2. Analytical Statement of Work

Analytical Services Coordinator

Write the analytical Statement of Work that communicates laboratory requirements from the ER project to the analytical laboratory.

3. Implementing Verification and Validation

   Verifier and Validator

   Implement verification and validation by completing the forms (Appendixes A and B, respectively).

B. Custody of Samples and Sample Documentation

The chain of custody (COC) is relevant to the representativeness of the sample to its origin. It is also legal documentation of sample possession. The COC documents the personal accountability of the samples, and any break in that signature series could result in jeopardizing the legal admissibility of the sample data.

While verification/validation cannot assure a sample has truly been in custody from the field to the final result, an evaluation of field and laboratory COCs will provide the best indicator. A sample is in custody if any of the following conditions are met:

- it is within one's possession;
- it is within one's view, after being in one's possession;
- it was in one's possession and then was secured to prevent tampering;
- it is placed in a designated secure area.

Data Verifier

1. Verification

   Verification includes CLP Form I header checks for accuracy from the COC. If header information is incorrect when compared to the COC, the verifier will mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, every attempt should be made to verify the true identity of each sample, else sample representativeness is in doubt.

   Data Verifier

   Trace the chain of signatures of all samples in the reporting batch from field sampling through receipt at the laboratory. If there is a break in the signature chain, indicate this problem in the verification report.

   Verify the headers of sample results summary reporting forms against COCs for accuracy. If header information is incorrect when compared to the COC, mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, make every attempt to verify the true identity of each sample; however, if sample representativeness remains in doubt, note the occurrence on the verification report.

2. Validation

   Data Validator

   Qualify sample data that is not traceable through signature records on COCs "R."

C. Holding Time and Turn-around Time

Holding times are defined as follows for volatile organics analysis:

- elapsed time between collection and analysis

Holding times are defined as follows for semivolatile organics analysis:

- elapsed time between collection and preparation, and between preparation and analysis

Holding times have been established by EPA to provide a maximum period of time in which an environmental sample will remain representative of its sampling location. Holding times begin when a sample is collected in the field, rather than the date of sample receipt at the laboratory, and ends with extraction and/or analysis.

If the final reported data results from a dilution or reinjection of the sample, this analysis must have been completed within the holding time. Laboratory preparation and analysis logs should be referenced to confirm holding times.

1. Deliverables

   - Field chains of custody
   - Laboratory chains of custody
   - Dates of collection, preparation and analysis for all samples

2. Verification

   Turn-around time is evaluated as **strictly a contractual issue**. An agreement should be established between the client and the laboratory concerning turn-around times for reported data. If turn-around times are not satisfied, a contractual mechanism should provide for appropriate action.

   Data Verifier

   Verify the presence of the pertinent chains of custody. Verify that sample results have been reported within the contractually-specified turn-around time. If data are noncompliant, note this in the verification report.

3. Criteria

   The following table indicates holding time requirements for the volatile and semivolatile (extractable) fractions. If preservation can not be verified the sample(s) must be considered unpreserved and qualified accordingly.

Holding time Criteria

Parameter	Matrix	Maximum Allowable Holding Time	Container	Preservative
Volatile Organics	Water	14 days**	Glass w/ teflon-lined septum	4°C; pH<2 with HCl, H_2SO_4, or solid $NaHSO_4$
	Soil	14 days	CLP: Glass, or closed-end tube (e.g. brass sleeve) SW-846 Low concentration (SW-5035)*: Glass with septum seal SW-846 High concentration (SW-5030)*: Glass with septum seal	4°C 4°C; pH≤2 with $NaHSO_4$ 4°C; Methanol
Extractable Organics	Water	7 days to extraction 40 days to analysis	Glass	4°C
	Soil	14 days to extraction 40 days to analysis	Glass	4°C

* SW-846 Update III provides several options for collection and preservation of solid samples for volatile organic compounds. Refer to Table 4-1 of SW-846 Update III and the applicable SW-846 preparation method for more information.

** For unpreserved water samples, the holding time is 7 days.

All samples in aqueous or soil matrices must be preserved by storage at temperatures of 4°C ± 2°C. Aromatic compounds in aqueous matrices must be acidified to pH < 2.0.

4. Validation

Data Validator

a. Qualify detected volatile/semivolatile compounds (VOCs/SVOCs) "J" and nondetected VOCs/SVOCs "UJ" if holding time requirements are not satisfied, unless qualified differently following the guidance below.

b. If holding times are grossly exceeded (e.g., by a factor of 2 or greater), use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. Should qualification be deemed necessary, detected VOCs/SVOCs may be considered estimated values and qualified "J", and nondetected VOCs/SVOCs may be qualified unusable,"R".

c. Qualify detected aromatic VOCs "J" and nondetected aromatic VOCs "UJ" if chemical preservation has not occurred or sample temperature has been excessive for aqueous samples. Other QC and raw sample data should be consulted to determine whether excessive temperatures and/or nonpreservation have contributed to significant loss of nonaromatic VOCs. Qualify detected nonaromatic VOCs "J" and nondetected nonaromatic VOCs "UJ" if it is determined that excessive temperatures and/or nonpreservation has affected sample data significantly.

D. Calibration of the GC/MS

1. GC/MS Performance Check (Volatile and Semivolatile organic compounds)

The instrument performance check is performed to ensure correct detection of ion abundance, mass resolution, identification, and to some degree, instrument sensitivity. These criteria are instrument-specific and not sample-specific; conformance is determined using standard material, therefore, these criteria should be satisfied in all circumstances.

a. Deliverables

The minimum deliverables necessary to complete the checks are described in this section:

- CLP Form V, or equivalent for SW-846 methods, for each GC/MS system used.
- Raw data for each instrument performance check for each GC/MS system used (required for confirmation).

b. Frequency

The instrument performance check must be analyzed at the beginning of each 12-hour period during which samples and/or standards are analyzed. If different instruments are used on samples in a similar case, the performance check(s) must be analyzed at this frequency as well.

c. Performance Criteria

The following tables present ion abundance criteria for both CLP and SW-846 methods.

Volatile Organic GC/MS Tuning Criteria

m/z	ION Abundance criteria (CLP SOW 3/90 – OLM03.1)	ION Abundance criteria (SW-846 Method 8260B)
50	8.0 - 40.0% of m/z 95	15.0 - 40.0% of m/z 95
75	30.0 - 66.0% of m/z 95	30.0 - 60.0% of m/z 95
95	Base peak, 100% rel. abundance	Base peak, 100% rel. abundance
96	5.0 - 9.0% of m/z 95	5.0 - 9.0% of m/z 95
173	< 2.0% of m/z 174	< 2.0% of m/z 174
174	50.0 - 120.0% of m/z 95	> 50.0% of m/z 95
175	4.0 - 9.0% of m/z 174	5.0 - 9.0% of m/z 174
176	93.0 - 101.0% of m/z 174	> 95.0%, but < 101.0% of m/z 174
177	5.0 - 9.0% of m/z 176	5.0 - 9.0% of m/z 176

Semivolatile Organic GC/MS Tuning Criteria

m/z	ION Abundance criteria (CLP SOW 3/90 – OLM03.1)	ION Abundance criteria (SW-846 Method 8270C)
51	30.0 - 80.0% m/z 198	30.0 - 60.0% of m/z 198
68	< 2.0% of m/z 69	< 2.0% of m/z 69
69	m/z 69 rel. abundance	m/z 69 rel. abundance
70	< 2.0% of m/z 69	< 2.0% of m/z 69
127	25.0 - 75.0% of m/z 198	40.0 - 60.0% of m/z 198
197	< 1.0% of m/z 198	< 1.0% of m/z 198
198	Base Peak, 100% rel. abundance	Base Peak, 100% rel. abundance
199	5.0 - 9.0% of m/z 198	5.0 - 9.0% of m/z 198
275	10.0 - 30.0% of m/z 198	10.0 - 30.0% of m/z 198
365	> 0.75% of m/z 198	> 1.00% of m/z 198
441	Present, but < m/z 443	Present, but < m/z 443
442	40.0 - 110.0% of m/z 198	> 40.0% of m/z 198
443	15.0 - 24.0% of m/z 442	17.0 - 23.0% of m/z 442

d. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

Data Validator

(1) Place reason code "P05" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "P06" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

(2) If mass assignment is in error (such as m/z 199 is indicated as the base peak for SVOC analysis, rather than m/z 198), qualify all detected and nondetected compounds as unusable (R).

(3) The following ion-abundances must always be satisfied:

Qualify data based on the specific circumstance of nonconformance to ion abundance criteria.

(a) Volatile ion abundances: m/z 95/96, 174/175, 174/176, 176/177

(b) Semivolatile ion abundances: m/z 198/199, 442/443, 68, 70, 197, 441

Raw data should be consulted to determine if associated sample and QC data can be considered usable or unusable if criteria for critical or non-critical ion-abundances are not met. Guidance to aid the application of evaluation of semivolatile compounds is as follows:

(4) m/z 198/199 and 442/443 are based on the natural abundances of ^{12}C and ^{13}C and should always be met.

(5) m/z 68, 70, 197, and 441 indicate the condition of the instrument and the suitability of resolution.

(6) m/z 365 is an indicator of suitable instrument zero adjustment. If m/z is zero, minimum detection limits may be affected. If m/z 365 is present but < 0.75%, the deficiency is not as serious.

(7) Other ion abundances are of minor importance.

Raw Data Confirmation

Confirm one of the ion abundance summaries from raw data. Inspect raw data to ensure that during the instrument performance check, three scans (apex, and scans immediately preceding and following the apex) have been averaged, and that a scan no more than 20 scans preceding the elution of BFB or DFTPP is used for background subtraction.

2. Initial Calibration of the GC/MS

Calibration is established to ensure that the instrument(s) is capable of producing acceptable qualitative and quantitative data and that the instrument(s) is capable of producing accurate and precise measurements based on known standard concentrations.

a. Deliverables

The minimum deliverables necessary to complete the checks are described in this section:

- CLP Form VI, or equivalent for SW-846 methods, for each initial calibration for each GC/MS system used.
- Raw data for each initial calibration for each GC/MS system used (required for confirmation).

b. Frequency

Initial calibration must be performed within 12 hours of the instrument performance check and prior to sample analysis.

c. Performance Criteria

On-Column Standard Concentrations and Performance Criteria for Initial Calibration

CLP Organic SOW 3/90 OLMO3.1	SW-846 Method 8260B (VOA) Method 8270C (SVOA)
10, 20, 50, 100, 200 ug/L VOA 20, 50, 80, 120, 160 ug/L SVOA minimum RRF of ▯ 0.05, %RSD ≤ 30.0%	5 levels minimum, lowest near but> MDL SW-8260B – Mean RF for SPCCs: ≥0.3 for chlorobenzene and 1,1,2,2-tetrachloroethane ≥0.1 for bromoform, chloromethane, and 1,1-dichloroethane SW-8270C – Mean RF for SPCCs: ≥0.05 SW-8260B and –8270C: %RSD for CCC ≤30%

d. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

Data Validator

(1) Place reason code "C07" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "C06" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

The following provides guidance for qualification of samples due to failure to satisfy minimum response factors or due to exceedence of maximum %RSD criteria. If initial RRF of any compound is <0.05 in the initial calibration, but is ▯ 0.05 in the continuing calibration, samples analyzed under that continuing calibration should be qualified due to the deficiency in initial calibration, because the linearity of the instrument has not been established within acceptance criteria.

(2) If the RRF (CLP) or RF (SW-846) of any compound is < 0.05
- qualify detects with positive mass spectral identification "J"
- qualify nondetects "R"

(3) If the %RSD among the calibration points is > 30%, and the RRF/RF is >0.05
- qualify detects "J"
- if upon inspection of raw data, nondetects are suspected to be reported falsely, qualify those data "UJ" or "R"

(4) If both the RRF/RF < 0.05 and %RSD >30%
- qualify detects "J"
- qualify nondetects "R"

(5) If the %RSD is > 30%, this indicates non-linearity in the calibration curve. Elimination of either the highest or lowest point in the curve may restore the %RSD. Recalculate the %RSD excluding the highest point and then excluding the lowest point.

(a) If elimination of either point does not restore %RSD < 30%
- qualify detects "J"
- qualify nondetects which are suspected to be false negatives "UJ" or "R"

(b) If elimination of the high point restores %RSD < 30%
- qualify detects outside linear portion "J"
- nondetects do not need qualification

(c) If elimination of the low point restores %RSD < 30%
- qualify low-level positive results in the area of non-linearity "J"
- qualify non-detected semivolatile target compounds using professional judgement

(6) If different matrices are included in the same SDG, verify that the correct initial calibration was used with each set of samples of similar matrix.

(7) If samples are quantitated against the initial calibration, ensure that the 50 µg/L (VOA) and 80 µg/L (SVOA) calibration runs are used for quantitation of sample results.

(8) Raw Data Confirmation

Raw data must be consulted before qualifying based on Initial Calibration results alone. Checks must be made for saturation, baseline shift, peak splitting, and other obvious interferences; if these interferences exist, document them in the validation report and qualify sample appropriately.

Raw data should be inspected for instances of manual integrations of peak areas. Reoccurring manual integrations on similar peaks within a calibration, manual integrations on peaks with normally good symmetry, and peak splitting manual integrations should be inspected to determine the necessity for integration, or if a systemic problem is occurring in the analyses.

Confirm the quantitation ions of two compounds in the initial calibration to determine whether the correct quantitation ions have been used to quantify the compounds. If incorrect ions have been shown, rationale should be provided in the data package for the non-compliance.

Equations G1 and G2 for calculating RRF and %RSD, respectively, are found in Attachment H.

3. Continuing Calibration:

a. Deliverables

- CLP Form VII, or equivalent for SW-846 methods, for each GC/MS system used.
- Raw data for each continuing calibration for each GC/MS system used (required for confirmation).

b. Frequency

Calibration is verified for VOA and SVOA once per 12 hour period in which samples are analyzed. The continuing calibration standard must be analyzed prior to sample analysis.

c. Performance Criteria

The following table lists performance criteria for continuing calibration.

Performance Criteria for Continuing Calibration

CLP Organic SOW 3/90 – OLMO3.1	SW-846 Method 8260B (VOA), Method 8270C (SVOA)
50 µg/L Minimum RRF of ▯ 0.05, %D < 25% from initial calibration	Mid-level standard – run every 12 hours SW-8260B – Mean RF for SPCCs: ≥0.3 for chlorobenzene and 1,1,2,2-tetrachloroethane ≥0.1 for bromoform, chloromethane, and 1,1-dichloroethane SW-8270C – Mean RF for SPCCs: ≥0.05 SW-8260B and –8270C: %D for CCC ≤ 20% from initial calibration

d. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator.

e. Validation

Data Validator

(1) Place reason code "C07" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "C06" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.
(2) If the %D exceeds ±25%, and the RRF is ≥0.05
- qualify detects "J"
- qualify nondetects "UJ"

(3) If the RRF is < 0.05
- qualify detects with acceptable mass spectral identification "J"
- qualify nondetects "R"

(4) If both the RRF < 0.05 <u>and</u> %D exceeds ±25%
- qualify detects "J"
- qualify nondetects "R"

(5) Raw Data Confirmation - Confirm the quantitation ions of two compounds in the continuing calibration to determine whether the correct quantitation ions have been used to quantify the compounds. If incorrect ions have been shown, rationale should be provided in the data package for the non-compliance.

E. Method Blank

All blanks (method, instrument, field, trip and equipment blanks) associated with the case must be evaluated against the sample results in the SDG. However, the validator should only apply qualification to those samples directly related to the affected blank (if more than one method blank is used per case). This section applies to target compounds and tentatively identified compounds.

1. Deliverables

The minimum deliverables necessary to complete the checks are described in this section:

- CLP Form I, CLP Form IV or equivalent for SW-846 methods, for each method blank.
- Raw data for each method blank (required for confirmation).

2. Frequency

Method Blank Preparation and Analysis Frequencies

CLP Organic SOW - 3/90 OLMO3.1	SW-846 Method 8260B (VOA) Method 8270C (SVOA)
· VOA - Method blank analyzed once per 12 hours · SVOA - once per case, each 14 day period during which samples in a case are received, each 20 samples in a case, or whenever samples are extracted by the same procedure	· Method blank analyzed for each batch (maximum of 20 samples/batch)

3. Performance Criteria

Compounds detected in the method blank must be less than the CRQL. Method blank performance criteria is not specified for SW-846 methods. For the purposes of validation, sample results shall be evaluated against CLP guidelines.

For volatile analyses, the method blank must contain < 2.5x CRQL for methylene chloride, <5x CRQL for acetone and 2-butanone, and ▯ CRQL for all other target compounds.

For semivolatile analyses, the method blank must contain < 5x CRQL for phthalate esters and ▯ CRQL for all other target compounds.

4. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

Data Validator

a. Place reason code "B07" on the affected data if non-correctable deliverable deficiencies have occurred; qualify only if the deviation indicates an adverse effect on data quality.

b. Any compound that is reported in both blank and sample must be evaluated; however, if the same compound is reported in sample(s) and more than one blank, the sample should be evaluated against the blank with the highest concentration of the compound. Sample results must not be modified by subtracting blank values. When comparing blank results to analytical samples results, ensure that factors such as dilution and different sample weights have been taken into consideration.

c. If sample concentration > CRQL and > 5* X Blank concentration
   - no qualification of result is necessary

d. If sample concentration is > CRQL and < 5* X Blank concentration
   - qualify the reported result with "U"

e. If sample concentration < CRQL and < 5* X Blank concentration
   - report SQL** and qualify the SQL with "U"

f. If gross contamination (saturated peaks in blank) is present
   - qualify all affected results "R"

* For the common laboratory artifacts - methylene chloride, acetone, and 2-Butanone (VOA); phthalate esters (SVOA) - use a factor of 10.

** See section VII.J concerning Sample Quantitation Limits

If the reviewer can determine where contamination originated other than through method blank contamination, an explanation must be presented in the validation report, and sample data will be qualified appropriately.

If inordinate numbers of other target compounds are found at low levels in the blank(s), it may be indicative of a systemic laboratory problem, and should be mentioned in the validation report.

An instrument blank must be analyzed following the analysis of an analytical sample showing saturated signals. If this is not done, the validator must evaluate the analyses following the saturated sample analysis for carryover. Qualify reported compounds significantly affected by instrument carryover "J" or "R."

g. Raw Data Confirmation

Determine from raw data whether compounds reported in the method blank are detected above the CRQL.

F. Surrogate Standards (System Monitoring Compounds):

Surrogate standards are non-target compounds added to all analytical samples, blanks and QC samples to assess overall system performance. These standards are added prior to GC/MS purge in water samples analyzed for volatiles, and before extraction in soil samples analyzed for volatiles and soil and water samples analyzed for semivolatiles.

1. Deliverables

- CLP Form II, or equivalent for SW-846 methods, including surrogate recoveries for all samples, blanks, and QC samples.
- Raw data for all samples, blanks, and QC samples (required for confirmation).

2. Frequency

Surrogate standards are added to all analytical samples, blanks and QC samples.

3. Performance Criteria

Volatile Surrogate Recovery Limits.

COMPOUND	WATER		SOIL	
	OLM03.1	SW-846 Method 8260B	OLM03.1	SW-846 Method 8260B
Toluene-d8	88 -110	88 – 110*	84 - 138	81 – 117*
Bromofluorobenzene	86 - 115	86 – 115*	59 - 113	74 – 121*
1,2-Dichloroethane-d4	76 – 114	80 – 120*	70 – 121	80 – 120*
Dibromofluoromethane	not required	86 – 118*	not required	80 – 120*

* For method SW-8260B, compare surrogate %R to laboratory-established limits. If no laboratory limits are available, these limits may be used.

Semivolatile Surrogate Recovery Limits.

COMPOUND	WATER		SOIL	
	OLM03.1	SW-846 Method 8270C	OLM03.1	SW-846 Method 8270C
Nitrobenzene-d5	35-114	Lab-determined limits**	23-120	Lab-determined limits**
2-Fluorobiphenyl	43-116	Lab-determined limits**	30-115	Lab-determined limits**
Terphenyl-d14	33-141	Lab-determined limits**	18-137	Lab-determined limits**
Phenol-d5	10-110	Lab-determined limits**	24-113	Lab-determined limits**
2-Fluorophenol	21-110	Lab-determined limits**	25-121	Lab-determined limits**
2,4,6--Tribromophenol	10-123	Lab-determined limits**	19-122	Lab-determined limits**
2-Chlorophenol-d4	33-110*	not required	20-130*	not required
1,2-Dichlorobenzene-d4	16-110*	not required	20-130*	not required

* Advisory Limits

** Surrogate control limits are developed by the laboratory. If laboratory-determined limits are wider than CLP limits, CLP criteria shall be used.

Concentrations of Surrogate Standards

Matrix	Fraction	Surrogate Standard Concentration
Water Soil	VOA	50 ▯g/L 50 ▯g/Kg
Water/Soil	Base and Neutrals	50 ng/▯L
Water/Soil	Acids	75 ng/▯L

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms, the identity of the proper surrogates and surrogate concentrations; if the required reporting forms or required surrogates or concentrations are not used, and these instances are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the instance should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "S06" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "S05" if inappropriate surrogate concentrations have been used; and place reason code "S04" if inappropriate surrogate standards have been used; qualify only if the deviation indicates an adverse effect on data quality.

   b. Qualify data if either of the following conditions are met: (1) one or more VOA surrogates is out of criteria, or (2) two or more base, neutrals or two or more acid surrogates are out of criteria. Professional judgement must be used if recovery is suspected to be effected by matrix interferences.

      (1) If the surrogate %R exceeds the upper control limit
         - qualify detects "J"
         - no qualification is necessary for nondetects

      (2) If surrogate %R is between 10% and the lower control limit
         - qualify detects "J"
         - qualify nondetects "UJ"

      (3) If surrogate %R is less than 10%
         - qualify detects "J"
         - qualify nondetects "R"

c. Reanalyses of samples must be inspected to determine which analysis provides the best results. The choice must be based on at least the following criteria:

- Surrogate percent recoveries
- Holding times
- Comparison of concentration of target compounds
- Internal standard areas and retention times

d. Surrogates in Method Blanks

Method blanks must have no surrogate standards out of criteria. The basic concern is whether the method blank problems represent an isolated problem with the blank alone, or whether there is a fundamental problem with the analytical process. If a method blank shows surrogates outside percent recovery criteria, the method blank should have been reanalyzed. If reanalysis has been done, and the method blank still shows surrogates outside criteria, the entire batch of samples associated with the non-compliant blank must be re-extracted (not applicable to purge-and-trap VOA). If the laboratory has not made the effort to take corrective action concerning non-compliant method blanks, this should be noted in the case narrative, and sample data should be qualified appropriately. If all samples and blanks show noncompliant surrogate recoveries, this indicates a systematic problem, and all sample data should be qualified. If just the blank(s) shows noncompliant recovery, this may be an isolated problem, and sample data qualification may not be necessary.

G. Internal Standards

Internal standards are used to ensure that GC/MS sensitivity and response are stable during analysis.

1. Deliverables

- CLP Form VIII or equivalent for SW-846 analyses
- Raw data for all samples, blanks, and QC samples (required for confirmation).

2. Frequency

Internal standards are added to all analytical samples, blanks, and quality control samples prior to purging for VOA water and soils, and are added prior to extraction of SVOAs and VOA medium level soils.

3. Performance Criteria

a. Retention Time Criteria

Sample internal standard retention times should not vary by more than ± 30 seconds, from the retention time of the associated continuing calibration standard.

b. Peak Area Criteria

Sample internal standard peak areas shall not vary by more than a factor of 2 (-50% to +100%) from the continuing calibration internal standard peak areas.

c. Concentrations

Concentrations of Internal Standards

Matrix	Fraction	Internal Standard Concentration
Water Soil	VOA	50 µg/L 50 µg/Kg
Water/Soil	Base/Neutrals	20 ng/µL
Water/Soil	Acids	20 ng/µL

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms, the identity of the proper internal standards and concentrations; if the required reporting forms or required internal standards or concentrations are not used, and these instances are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the instance should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code I07 on the affected data if non-correctable deliverable deficiencies have occurred; qualify only if the deviation indicates an adverse effect on data quality.

   b. The following provides guidance for qualification of samples due to poor internal standard performance. Resulting qualification of compounds is based on results for the associated internal standard.

      - If peak area %D is either <-50% or > 100%
        - qualify detects "J"
        - nondetects require no qualification
      - If peak area is < 50%
        - qualify nondetects "UJ" and report SQL
      - If a performance drop is indicated by extremely low area counts
        - qualify detects "J" or "R" if the performance drop has significantly affected sample results
        - qualify nondetects "R"

      For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction; however, sample results should not need to be rejected if mass spectral criteria are met.

c. Raw Data Confirmation

If Internal Standard retention times vary by more than ± 30 seconds, (between the sample internal standard and calibration internal standard), examine the chromatographic profile for that sample to determine if any false positives or negatives exist if raw data exists for the sample in question. Qualify false positive results or false negative detection limits "R" as appropriate.

H. Matrix Spike / Matrix Spike Duplicates (MS/MSD)

The purpose of the MS/MSD differs from the CLP 3/90 SOW to the SW-846 methods. For CLP, the MS/MSD are analyzed to determine long-term accuracy and precision of the analytical method on various matrices using target compounds. For SW-846, the MS/MSD are analyzed to determine the accuracy of the method. However, if recovery criteria are not satisfied for the SW-846 methods, there is difficulty in assessing whether the cause was the method or matrix-related interferences.

MS/MSD data alone should not be used exclusively to qualify data, but in conjunction with other supporting QC data. Professional judgement should be used in qualifying samples data based on based on MS/MSD percent recovery and precision data.

1. Deliverables

- CLP Form III or equivalent for SW-846 Methods
- Raw data for each matrix spike and matrix spike duplicate (required for confirmation).

2. Frequency

MS/MSD are analyzed at a frequency of once per 20 samples of similar matrix (whichever is more frequent), and concurrently with the samples in the SDG.

3. Performance Criteria

Matrix spike %R for compounds analyzed under SW-846 method 8270C shall be compared to laboratory-established limits. If laboratory limits have not been provided or have not been established, multilaboratory performance data located in Appendix H may be used.

For SW-846 method 8260B, the MS/MSD percent recovery should lie within the QC acceptance criteria determined from the analysis of laboratory control samples during method validation.

The following tables list CLP performance criteria for MS/MSD.

CLP Performance Criteria for VOA MS/MSD

VOA Compound	Water Percent Recovery	Water Percent Difference	Soil Percent Recovery	Soil Percent Difference
1,1-Dichloroethene	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

CLP Performance Criteria for SVOA MS/MSD

SVOA Compound	Water Percent Recovery	Water Percent Difference	Soil Percent Recovery	Soil Percent Difference
Phenol	12-110	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-97	28	28-104	27
N-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39-98	28	38-107	23
4-Chloro-3-methylphenol	23-97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10-80	50	11-114	50
2,4-Dinitrotoluene	24-96	38	28-89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

For SW-846 methods, the MS/MSD recovery should fall within laboratory-determined limits. If MS/MSD results fall outside the laboratory-determined limits (or limits provided in Appendix H for SVOCs), a QC Check Standard or Laboratory Control Sample must be analyzed and fall within those ranges.

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "M05" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "M04" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

   b. A determination shall be made concerning what extent that non-compliant MS/MSD data has on other sample data in regard to the MS/MSD sample itself as well as specific compounds in samples associated with the MS/MSD. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to that sample alone. However, it may be determined that the laboratory is having a systematic problem in the analysis of one or more compound, which affects all associated samples. Professional judgement will be used to determine the need for "J" qualification of positive results of non-spiked compounds.

I. QC Check Standard

The QC Check Standard is not required for the CLP 3/90 SOW but is required for the SW-846 methods 8260B and 8270C. Four replicates of the QC check standard are extracted and analyzed as an initial, one-time demonstration of ability to generate acceptable accuracy and precision. The QC check standard procedure may need to be repeated if changes in instrumentation or methodology occur. A QC check standard must also be analyzed if MS/MSD results fall outside laboratory-determined limits (or ranges designated in Appendix H for semivolatile analysis).

It is recommended that this standard be the same matrix as the analytical samples, and for semivolatile analysis prepared and analyzed with the batch of analytical samples (although the SW-846 requires analysis only). Unless prepared with the analytical samples, the QC Check Standard will not provide a representation of method accuracy. The QC Check Standard is prepared from addition of a QC Check Standard concentrate into the appropriate clean matrix, extracted and analyzed (analyzed only in the case of volatile purge and trap). Only those compounds that failed the MS/MSD criteria need to be reported. Acceptance criteria for QC Check Standard for SVOCs is provided in Appendix G.

1. Deliverables

   - Report summary of all analytes out of compliance in MS but reported in QC Check Standard
   - Raw data for each QC Check Standard (required for confirmation).

2. Frequency

   The QC Check Standard shall be analyzed when the MS/MSD recovery exceeds the ranges specified in the respective analytical SW-846 method.

3. Performance Criteria

   The QC Check Standard must be analyzed and must fall within laboratory-determined limits, or ranges specified in the method.

4. Verification

   Data Verifier

   Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

5. Validation

   Data Validator

   a. Place reason code "K04" on the affected data if non-correctable deliverable deficiencies have occurred; qualify only if the deviation indicates an adverse effect on data quality.

   b. Confirm that the QC Check Standard was analyzed (VOA) or prepared and analyzed (SVOA) if the MS/MSD percent recovery criteria were not satisfied. If the volatile QC Check Standard was analyzed or if the semivolatile QC Check Standard was prepared and analyzed and the same noncompliant compounds in the MS/MSD exceed criteria, qualify those compounds in the affected samples "J," using professional judgement to qualify "R."

   c. If the semivolatile QC Check Standard was analyzed only (i.e. not prepared) it will provide limited value for method accuracy. Qualification should be applied only if the QC Check Standard and other QC data within the batch indicate that the accuracy of reported compounds has been affected.

J. CRQLs and SQLs

CRQL(s) have been developed to enable the laboratory to meet realistic detection limit goals. CRQL(s) have been determined by EPA to be calculated as 3 above the MDL.

Due to deviations from method-specified sample weights, extract volume or aliquot used in analysis, or due to dilution or soil percent moisture, CRQLs are modified accordingly and are termed "sample quantitation limits" (SQL).

1. Deliverables

   - CLP Form I or equivalent for SW-846 analytical methods for all samples

2. Frequency

CRQLs or SQLs are reported for all compounds that are not detected above the method MDL.

3. Verification

Data Verifier

For all samples, the SQL must be less than the Required Detection Limit (RDL), which are identified and communicated to the laboratory in the laboratory SOW. If the SQL is greater than the RDL, this may indicate matrix-related problems or analytical conditions precluding RDL achievement.

All sample results shall be reviewed to determine RDL compliance. In cases where the SQL is greater than the RDL, the project may decide to request a reanalysis.

Verify that compounds detected at levels below the SQL have been qualified "J" by the laboratory.

4. Validation

Data Validator

a. For one non-detected compound in each sample and blank, verify that CRQL(s) have been adjusted for deviations from the nominal preparation and analysis conditions such as sample size, aliquot if necessary.

b. No additional validation qualifiers are necessary for results detected below the SQL unless directed in other sections of this document.

c. Calculations for modifications to the CRQL can be found in Appendix F.

K. Target Compound Identification:

Compound identification is the step in the validation process that involves verifying the identity and concentration of the target compounds.

1. Deliverables

- CLP Form I or equivalent for SW-846 analytical methods
- Raw data for each sample

2. Performance Criteria

Mass spectra of the sample target compound and a current laboratory-generated standard must match according to the following criteria:

a. All ions at > 10 % relative intensity in the standard reference spectrum must be present in the sample mass spectrum. All ions > 10 % relative intensity in the sample mass spectrum but not in the standard mass spectrum must be considered and accounted for.

b. The relative intensities of the ions must agree within ±20% between the standard and sample spectrum.

c. The relative retention time (RRT) of the target compound must be within ± 0.06 units of the standard RRT.

3. Validation

Data Validator

a. Raw Data Confirmation

The presence/absence and concentration of detected compounds in the samples are reviewed to determine whether or not the correct quantitation ions have been used for proper quantification of the compounds. If incorrect ions have been shown, rationale should be provided in the data package for the non-compliance. If no rationale has been provided, evaluation of the effect on quantitation of detected target compounds should be made. If detected target compounds quantitated against the incorrect ion are significantly affected, the affected compounds may be qualified "R."

The validator should inspect the data for instances of manual integrations of peak areas. Reoccurring manual integrations on similar peaks from sample to sample or from calibration to sample, or on peaks with normally good peak resolution , or for splitting of peaks should be inspected to determine the necessity for integration, or if a systemic problem is occurring in the analyses.

Situations that may tend to produce carryover to subsequent sample analyses, such as the analysis of samples showing high concentrations of compounds, shall be evaluated. If cross-contamination has had an effect on a compound, such as reporting of false positives, or artificially elevating compound levels, affected data may be qualified "R."

Samples are diluted and reanalyzed if compound signals exceed the dynamic range of the instrument (saturation) or if interferences preclude accurate quantitation of compounds. When a sample is reanalyzed and both analyses of that sample are included in the data package, indicate on the laboratory reporting forms which results are the most reliable.

L. Manual Recalculation of Analytical Results

The accuracy and consistency of sample result calculation by the laboratory can be addressed by two different techniques. The application of each strategy depends on the laboratory's ability to minimize transcription during reporting, and how familiar the project is with the performance of the laboratory. If sample results are produced primarily through software processing and minimal transcription is performed in the laboratory, the data system(s) can be evaluated during an audit or surveillance by performing two different tests on the software (1) supply the data system a consistent set of input designed to provide a consistent set of output, and (2) supply the data system a set of nonconforming data to test the error detection routines. An additional evaluation of the laboratory's software configuration control and security is also necessary. Through this technique, a high level of confidence can be gained in the laboratory's reporting techniques and will result in a minimal need for manual recalculation of sample results.

If the laboratory has a high rate of manual transcription in generation of sample results, the project may choose to manually recalculate sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contacting the laboratory may be necessary to resolve the problem.

1. Validation

Data Validator

Data may be qualified "R" as a last resort if no actions can reproduce reported values.

Calculations and rounding rules for compound quantitation can be found in Appendix F.

M. Tentatively Identified Compounds (TICs)

TIC are non-target compounds that are not system monitoring compounds or internal standards. TICs are qualitatively identified by using mass spectral identification from a mass spectra library search. The laboratory must identify the 10 largest VOA peaks and 20 SVOA peaks that are not surrogates, internal standards, or target compounds.

1. Deliverables

- CLP Form I-TIC for each sample or project-specified summary reports with the same information.
- Raw data for each sample

2. Verification

Data Verifier

Verify the presence of the pertinent reporting forms. If the required reporting forms are not present, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

3. Validation

Data Validator

a. Raw Data Confirmation

The validator should check raw data against TIC report ensuring that all TIC peaks are accounted on CLP Form I.

Two named TIC (not "unknowns") concentrations shall be recalculated using the calculations in Appendix F with a RRF of 1.0.

The following are guidance for identification/qualification of TICs:

(1) The validator shall qualify all TICs as NJ.
(2) Mass spectra for all samples with raw data, and blanks shall be examined for TICs.
(3) All ions > 10% relative intensity in the reference spectrum should be in the sample spectrum
(4) Relative intensity of the major ions should agree within ± 20% between sample and reference spectra.

(5) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(6) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or co-elution of additional TIC or target compounds.

(7) If the identification is uncertain or there are extenuating factors affecting compound identification the TIC result may be reported as "unknown."

(8) TICs less than 10 X the level in the blank should not be reported. If a TIC is reported at this level, qualify R.

(9) TICs may be reported as "either compound X or compound Y" if there are more than one reasonable match from the library search.

(10) All similar TICs may be reported as a total: (e.g. all alkanes may be reported as total hydrocarbons).

(11) If TIC evaluation from library search does not yield conclusive evidence from items stated above, the validator may change the identification of the TIC to "unknown." Professional judgement should be used in comparing references spectra to sample spectra and the incidence of TICs in multiple samples and blanks.

(12) The validator should be aware that common laboratory artifacts should not be reported as TICs. The validator shall qualify these compounds R if reported as TICs.

(13) If a TIC is reported in one or all samples but not in the blank, the validator should check if the compound is a common laboratory artifact in the sample, and inspect in the blank for peaks that are < 10% of the internal standard area but are present in the blank chromatogram at a similar retention time.

(14) Compounds reported as a TIC in one fraction should be qualified "R" in that fraction if that compound is reported as detected in another fraction.

(15) Blank chromatograms should be examined to verify that TIC peaks present in samples are not found in blanks. When a low-level non target compound which is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank chromatograms may require looking for peaks which are less than 10 percent of the internal standard area but present in the blanks chromatogram at similar relative retention time.

(16) If target compounds are identified by non-target library searches the laboratory should be contacted to resubmit the relevant forms with the target compound quantitated against the correct quantitation ion.

The following list presents the common laboratory contaminants that should not be reported as TICs in either VOA or SVOA fractions:

<u>Common Laboratory contaminants</u>
CO_2 (m/z 44), (may be introduced by system leaks)
siloxanes(m/z 73) (common GC column bleed artifacts)
diethyl ether (1,1-oxybisethane)
hexane
1,1,2-trichloro-1,2,2-trifluoroethane (fluorotrichloromethane or Freon 113)
phthalates at levels less than 100 ug/L (waters) or 4000 ug/Kg (soils)

<u>Solvent Preservatives and By-products</u>
cyclohexene
cyclohexanone
cyclohexenone
cyclohexanol

chlorocyclohexene
chlorocyclohexanol

Aldol Reaction Products of Acetone
4-methyl-2-penten-2-one
4-hydroxy-4-methyl-2-pentanone
5,5-dimethyl-2(5H)-furanone

VIII. REQUIRED RECORDS

A. General Records

Records arising from the actions in this document are as follows:

- The verification report.
- The validation report.
- Copies of qualified, or unqualified results reports.

These records become part of the analytical data package and must be controlled through a records management system.

B. Quality Assurance Records

This procedure is considered a Quality Assurance Record and, as such, a record copy will be provided to the appropriate document management center (DMC) for retention according to the minimum schedule identified by DMC personnel.

IX. ADMINISTRATION

The Project Manager or Watershed Manager is responsible for ensuring that the requirements in this document are incorporated into project verification/validation plans. The data validation coordinator is responsible for incorporating the requirements into project plans and for interfacing with the ER-Data Quality Program and with other members of the project in its interpretation.

X. APPENDIXES

Appendix A: Data Verification Report
Appendix B: Data Validation Report
Appendix C: Qualifiers and Reason Codes
Appendix D: Volatile and Semivolatile Data Validation Implementation Form
Appendix E: Qualification Tables for Multiple Quality Deficiencies
Appendix F: Calculations and Equations
Appendix G:Volatile and Semivolatile Matrix Spike Performance Criteria

APPENDIX A

Volatile/Semivolatile Data Verification Report

Sample Delivery Group #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW#: _____

This checklist is to be used for verification of VOA/SVOA data packages, consistent with the procedure ES-B-0810. No qualifiers are placed through the process of verification.

Verified by:_____ Date:_____

APPENDIX A

	Yes	No
Custody of Samples		
1. Are samples traceable through inspection of signature records on field and Laboratory chains of custody?	___	
Action: For any samples not traceable upon inspection of chains of custody, indicate the identities below and where the break in traceability occurred.		
GC/MS Performance Check		
2. Are Form V's or equivalent for SW-846 methods included in the data package?	___	___
3. Has the performance check been run at the specified frequency?	___	___
Calibration		
4 Are Forms VI and VII or equivalent for SW-846 methods included in the data package?	___	___
5. Has initial calibration been performed within 12 hours of the performance check and prior to analysis of analytical samples?	___	___
6. Has continuing calibration been performed every 12 hours in which samples were analyzed?	___	___
7. Have the contract-required standard concentrations been used (or those specified in SW-846)?	___	___
Surrogate Standards		
8. Are Form II's or equivalent for SW-846 methods included in the data package?	___	___
9. Are surrogate recoveries included for all samples, blanks, and QC samples?	___	___
10. Have the contract-specified surrogate standard concentrations been used?	___	___
Internal Standards		
11. Are Form VIII's or equivalent for SW-846 methods included in the data package?	___	___
12 Have the contract-specified internal standard concentrations been used?	___	___
Method Blank		
13. Are Form I's and Form IV's or equivalent for SW-846 methods, for method blanks, included in the data package?	___	___
14. Have frequency requirements for the method blank been satisfied?	___	___
Matrix Spike		
15. Are Form III or equivalent for SW-846 methods included in the data package?	___	___
16 Have frequency requirements for the MS/MSD been satisfied?	___	___
QC Check Standard (SW-846 only)		
17. Is a report for the QC Check Standard for SW-846 methods included in the data package?	___	___
18 Was the SVOA QC Check standard both prepared and analyzed?	___	___
CRQLs and SQLs		
19. Are Form I's or equivalent for SW-846 methods included in the data package?	___	___
20. Are all SQLs ▯ the RDL?	___	___
21. Are all compounds detected or not detected below the SQL qualified "J" by the laboratory?	___	___

APPENDIX A

Action:	Indicate non-correctable contract compliance deviations below. If SQL>RDL, reanalysis may be warranted. Specify compounds which will require "J" qualification as stated in #3 above.

	Yes	No
Tentatively Identified Compounds 22. Are Form I-TIC or equivalent for SW-846 methods for all analytical samples and blanks included in the data package?	___	___

Action:	Indicate non-correctable contract compliance deviations below.

_____

_____

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APPENDIX B

Volatile/Semivolatile Data Validation Report

Sample Delivery Group #: _____

Matrix: _____

Laboratory: _____

Laboratory SOW#: _____

This checklist is to be used for validation of VOA/SVOA data packages, consistent with the procedure ES-B-0810. This checklist should be used only if the VOA/SVOA Verification Report has been completed.

Instructions: If criteria are not met in this checklist, follow qualification provided in the "Q" column accompanied with the corresponding reason code in the "C" column. If necessary, additional codes may be placed. Sections titled "Raw Data Confirmation" need only be performed if necessary, as determined through the DQO process.

Validated by:_____ Date:_____

APPENDIX B

1.0 Custody of Samples

Validation Step
Action: For any samples not traceable upon inspection of verification report and chains of custody, indicate the identities below and where the break in traceability occurred. Indicate which samples were affected below.
Qualified Sample _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____ _____

APPENDIX B

2.0 Technical Holding Times and Sample Preservation

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Were all samples analyzed within the appropriate technical holding time?			J	UJ/R	H01, H02, H03, H04
2. Were all samples preserved properly?			J	UJ	H05

Action: Indicate affected samples below.

Sample	Technical Holding Time Exceedence	Improper Preservation
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

APPENDIX B

3.0 GC/MS Performance Check

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. The GC/MS performance check has been performed at the proper frequency.			*	*	T02
1 No misassignment of ions has occurred.			R	R	T01
2 All ions are within ion-abundance criteria.			J/R	J/R	T03
Raw Data Confirmation					
3 Raw data show proper averaging of scans.			**	**	T05

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Rejection
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

* Qualify only if the deviation indicates an adverse effect on data quality.

** Qualify as appropriate

APPENDIX B

4.0 Initial Calibration

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Initial calibration has been performed within 12 hours of the GC/MS performance check and prior to sample analysis.			*	*	C06
2 Are all compounds' RRF > 0.05?			J	R	C01
3 Are all compounds' RSD among calibration points <30%?			J	**	C02
4 Compounds with RRF<0.05 and %RSD >30% are qualified as follows.			J	R	C01, C02
6. Elimination of either high or low point restores %RSD<30%.			J***	n/a	C02
7. Samples with differing matrices have matching initial calibration matrix.			**	**	C10
8. Samples quantitated against initial calibration use 50 g/L (VOA) and 80 g/L (SVOA).			**	**	C11
Raw Data Confirmation					
9. Raw data have been examined for anomalies.			**	**	"Q" Codes
10. The quantitation ions of 2 compounds have been confirmed as the correct ions for quantitation.			**	**	
Action: Indicate affected samples only if data are rejected.					
Sample _____ Reason for Rejection _____					

* Qualify only if the deviation indicates an adverse effect on data quality.
** Qualify as appropriate
*** Qualify only peaks outside linear portion

APPENDIX B

5.0 Continuing Calibration

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Continuing calibration has been performed within 12 hours in which samples are analyzed.			*	*	C06
2 Are all compounds' RRF > 0.05?			J	R	C04
3. Are all compounds' %D between initial and continuing calibration points <25%?			J	UJ	C05
4 Compounds with RRF<0.05 and %D >25% are qualified as follows.			J	R	C04, C05

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Rejection

* Qualify only if the deviation indicates an adverse effect on data quality.

APPENDIX B

6.0 Method Blank

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Was the method blank analyzed at the appropriate frequency?			*	*	B05
2. Was the method blank the same matrix as the samples?					B02
3. Sample results are > CRQL and > 5** X Blank result					
sample result is > CRQL and < 5** X Blank result			U	n/a	B01
sample result is < CRQL and < 5** X Blank result			U (report SQL)	n/a	B01
gross contamination			R	n/a	B03
Raw Data Confirmation					
4. Confirm from raw data that compounds reported in the method blank are detected above the CRQL.					
Action: Indicate affected samples only if data are rejected.					
Sample	Reason for Qualification				
_____	_____				
_____	_____				
_____	_____				
_____	_____				
_____	_____				
_____	_____				
_____	_____				
_____	_____				

* Qualify only if the deviation indicates an adverse effect on data quality.

** 10x for common lab contaminants

APPENDIX B

7.0 Surrogate Standards

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Have the proper surrogate standards and surrogate concentrations been used?			*	*	S04, S05
2. %R is > upper control limit			J	n/a	S01
3. %R is between 10 and lower control limit			J	UJ	S02
4. If surrogate is <10% compounds are qualified as follows			J	R	S03

Action: Indicate affected samples only if data were rejected.

Sample	Reason for Qualification

* Qualify only if the deviation indicates an adverse effect on data quality.

APPENDIX B

8.0 Internal Standards

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Have the proper internal standards and internal standard concentrations been used?			*	*	I05, I06
2. Are peak areas between -50% and +100% of the continuing calibration internal standard peak areas?					
if < 50%			J	UJ	I02
if >100%			J	n/a	I01
extremely low area counts			**	R	I03
3. Internal standard retention time does not vary more than 30 seconds from continuing calibration.			J/R**	R**	I04

Action: Indicate affected samples only if data were rejected.

Sample	Reason for Qualification

* Qualify only if the deviation indicates an adverse effect on data quality.

** Qualify as appropriate

APPENDIX B

9.0 Matrix Spike/Matrix Spike Duplicate

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Was the MS/MSD analyzed at the appropriate frequency?			*	*	M04
2. Are all MS/MSD compounds within control criteria?			**	**	M01, M02
3. Are all MS/MSD RPD within control criteria?			**	**	M03

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

* Qualify only if the deviation indicates an adverse effect on data quality.

** Qualify only after evaluating other QC data in the SDG.

APPENDIX B

10.0 QC Check Standard (SW-846 methods only)

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Were all noncompliant compounds in the MS/MSD reported in the QC Check Standard?			J	UJ	K04
2. Was the QC Check Standard prepared and analyzed for SVOA compounds?			*	*	K01
2. Were the %R of the reported compounds within acceptance criteria?			J	UJ/R	K02, K03

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

* Qualify only if the deviation indicates an adverse effect on data quality.

APPENDIX B

11.0 Sample Data Evaluation

Validation Step
1. For one compound in each sample, determine whether CRQLs have been modifed to SQLs.
2. Sample-specific evaluation - these evaluations are performed one sample in each batch (excluding QC)
evaluate whether the correct quantitation ions were used
evaluate adequacy of manual integrations of peak areas.
evaluate the presence of carryover
evaluate the validity of dilutions and reanalyses
evaluate interferences which may preclude accurate quantitation of compounds.
3. Manually recalculate 2 detected and 2 nondetected results from a selected sample in the SDG.
4. Did recalculation confirm reported results? If not, increase the frequency of recalculations until adequate confidence is gained in the reported results.
5. Qualify all TICs "NJ"
6. Evaluate all TICs according to VIII.M.3 of the VOA/SVOA verification/validation procedure.
Action: Indicate instances of manual calculations not confirming reported results. Where samples have been reanalyzed, and both reanalyses are included in the data package, indicate on the laboratory reporting forms which results are the most reliable.
Description of Sample-specific Evaluation Problems _____ _____ _____ _____ _____

APPENDIX C

Data Validation Qualifiers and Reason Codes

Volatile and Semivolatile Analytical Data

Reason codes must be used with all qualifiers placed on analytical data.

U Compound not detected above the reported detection limit.

J Compound identified as detected; the associated numerical value is approximated.

UJ Compound not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.

NJ Presumptively present at an estimated quantity (use with TICs only).

R Result is not usable for its intended purpose.

= "Equals" sign, indicates that no qualifier is necessary.

DATA VALIDATION QUALIFICATION CODES

VOA and SVOA Analytical Data

Blanks

B01 Concentration of contaminant in the blank at a level ▯ the qualification level.
B02 Blank was not the same matrix as the analytical samples.
B03 Gross contamination exists.
B05 Blanks were not analyzed at appropriate frequency.
B07 Blank data not reported.
B09 Other (describe in comments).

APPENDIX C

Calibration

C01 Initial calibration RRF was <0.05.
C02 Initial calibration RSD was >30%.
C04 Continuing calibration RRF was <0.05.
C05 Continuing calibration %D was >25%.
C06 Calibration or performance check was not performed at the appropriate frequency.
C07 Calibration data not reported
C08 Calibration not performed.
C09 Chemical resolution criteria were not satisfied.
C10 Calibration standard matrix not the same as sample matrix
C11 Compounds quantitated against inappropriate standard or standard concentration level
C12 Compound quantitated against inappropriate ion
C14 Retention time of compound outside window.
C18 Inappropriate standard concentrations
C24 Standard curve was established with fewer than the appropriate number of standards
C31 Other (describe in comments)

Evidentiary Concerns

E01 Custody of Sample in Question.
E02 Standard not Traceable.
E03 Other (describe in comments)

General

G01 Professional judgement was used to qualify the data.
G02 Other (describe in comments)

Holding Times/Preservation

H01 Extraction holding times were exceeded.
H02 Extraction holding times were grossly exceeded.
H03 Analysis holding times were exceeded.
H04 Analysis holding times were grossly exceeded.
H05 Samples were not preserved properly.
H06 Sample preservation can not be confirmed
H07 Sample temperature exceeded criteria prior to preparation
H08 Other (describe in comments)

APPENDIX C

Internal Standards

I01 Area count was above upper control limits.
I02 Area count was below lower control limits.
I03 Extremely low area counts or performance was exhibited by a major drop off
I04 Internal standard retention time varied by more than 30 seconds
I05 Inappropriate internal standard used
I06 Inappropriate internal standard concentration(s) used
I07 Internal standard data not reported.
I08 Other (describe in comments)

QC Check Standard (Methods SW-8260B and 8270C)

K01 SVOA QC Check Standard not analytically prepared, but only analyzed
K02 Recovery of QC Check Standard was above upper control limits
K03 Recovery of QC Check Standard was below lower control limits
K04 QC Check Standard data not reported
K05 Other (describe in comments)

Matrix Spike and MS/MSD

M01 MS and/or MSD recovery above upper control limit.
M02 MS and/or MSD recovery below lower control limit.
M03 MS/MSD pair exceeds the RPD limit.
M04 MS and/or MS/MSD not analyzed at the appropriate frequency
M05 MS and/or MS/MSD data not reported
M06 Other (describe in comments)

Instrument Performance

P01 High background levels or a shift in the energy calibration were observed
P02 Extraneous peaks were observed.
P03 Loss of resolution was observed.
P04 Peak-tailing or peak splitting that may result in inaccurate quantitation were observed
P05 Instrument performance data not reported.
P06 Instrument performance not analyzed at the appropriate frequency
P07 Other (describe in comments)

APPENDIX C

Quantitation

Q01 Peak Misidentified
Q02 Target analyte affected by interfering peak.
Q03 Qualitative criteria were not satisfied.
Q04 Cross contamination occurred.
Q07 Analysis occurred outside 12 hr GC/MS window.
Q09 TIC result was not above 10 X the level found in the blank.
Q10 TIC Reported as Detect in Another Fraction.
Q11 Common Artifact Reported as a TIC.
Q12 No raw data were provided to confirm quantitation.
Q14 Inappropriate aliquot sizes were used.
Q18 Compounds were not adequately resolved.
Q24 Raw data anomaly
Q25 Other (describe in comments)

Surrogates

S01 Surrogate recovery was above the upper control limit.
S02 Surrogate recovery was below the lower control limit.
S03 Surrogate recovery was <10%.
S04 Inappropriate surrogate standard used
S05 Inappropriate surrogate standard concentration(s) used
S06 Surrogate data not reported.
S07 Surrogate outside retention window
S08 Other (describe in comments)

Instrument Tuning

T01 Mass calibration ion misassignment.
T02 Mass calibration was not performed every 12 hours.
T03 Mass calibration did not meet ion abundance criteria.
T04 Mass calibration data was not reported.
T05 Scans were not properly averaged
T06 Other (describe in comments)

APPENDIX D

Volatile and Semivolatile Data Validation Implementation Form

Purpose: This worksheet is the mechanism to be used to identify and document the implementation of procedure ES-B-0810.

Instructions: One worksheet must be completed for each fraction and analytical method (e.g. CLP Volatiles, SW-8260B Volatiles). The completed worksheet shall be forwarded to the ER Data Quality Program for approval.

If the requirements of ES-B-0810 are adopted as-is, without the necessity of approved alternatives, enter "as-is" for review item "A" in the implementation column and complete the project sign-off section at the bottom of the form. No other entries are needed.

If project-specific implementations are necessary,

1) Identify review items that will be directly implemented and enter "as-is" in the respective rows of the Implementation column.
2) Identify review items that will not be considered by verifiers or validators during the implementation of this procedure, and enter "n/a" in the respective rows of the Implementation column and reference attached materials documenting the reason for the exclusion of these review items in the Comment column.
3) Identify review items that will be implemented with project-specified alternative actions and enter "Alternative" in the respective rows of the Implementation column. In the Comment column, reference attached materials that document the need for the alternative actions and specify the actions to be implemented upon approval. This alternative description should cover the following: Deliverables, Frequency, Performance Criteria, Verification step(s), Validation step(s).
4) Complete the project sign-off section at the bottom of the worksheet and forward to the ER Data Quality Program for approval.

Fraction and Method:

		Implement	Comment
1.	All requirements of ERWM/ER-P2210 will be implemented.	____	_____
2.	Technical Holding Time	____	_____
3.	GC/MS Performance Check	____	_____
4.	Initial Calibration	____	_____
5.	Continuing Calibration	____	_____
6.	Surrogate Standards	____	_____
7.	Internal Standards	____	_____
8.	Method Blank	____	_____
9.	Matrix Spike/ Matrix Spike Duplicate	____	_____
10.	QC Check Standard	____	_____
11.	CRQL/SQL	____	_____
12.	Target Compound Identification	____	_____
13.	Manual Calculation of Results	____	_____
14.	Tentatively Identified Compounds	____	_____

The _____ project will approach volatile and semivolatile data validation with a strategy consistent with ES-B-0810, and/or with specific alternative(s) described on the attached pages.

_____ _____

Signature Date_

APPENDIX E

Guidance for Data Qualification

Due to Multiple Quality Deficiencies

This appendix provides guidance in the qualification of data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on potential effect on sample data. The effect of quality deficiencies may be applicable to only a single sample or to all samples within the reporting batch. A validation qualifier and associated reason code(s) should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

The following is a listing of data quality indicators and the probable effects on sample data.

Data Quality Indicator	Effect on Sample Data
GC/MS Tuning	compound identification
Initial calibration	identification and quantitation
Continuing calibration	quantitation
Surrogate standards	positive or negative bias
Internal standards	positive or negative bias
Method blank	positive bias
Matrix spike / matrix spike duplicate	positive or negative bias and precision
QC check standard	positive or negative bias

In the instance of multiple quality deficiencies the validation qualifier and reason code(s) should be placed consistent with the acceptable level of uncertainty associated with the intended use of the data. The validation SOW should provide a summary of the intended use(s) of the data. (e.g., risk assessment, fate and transport modeling, waste management) to facilitate appropriate placement of validation qualifiers.

APPENDIX F

Rounding rules

1. In a series of calculations, carry the extra digits through to the final result, then round off.

2. If the digit to be removed is less than 5, the preceding digit stays the same.

   If the digit to be removed is equal to or greater than 5, the preceding digit is increased by 1.

G.1 Calculation for RRF:

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

where:
- A_x = Area of the characteristic ion of the compound
- A_{is} = Area of the characteristic ion of the internal standard
- C_x = Concentration of the compound
- C_{is} = Concentration of the internal standard

G.2 Calculation for %RSD:

$$\%RSD = \frac{\sigma}{\overline{X}_{(R1,R2)}} x100$$

where:
- σ = standard deviation of the five initial calibration RRFs (per compound)
- $X_{(R1, R2)}$ = mean of the five initial calibration RRFs (per compound)

G.3 Surrogate Standard Concentration

$$C_{ss} = \frac{A_{ss} x I_s}{A_{is} x RRF_{50}}$$

where:
- C_{ss} = concentration of surrogate
- A_{ss} = area of surrogate
- I_s = concentration of Internal standard
- A_{is} = area of internal standard
- RRF = relative response factor (from continuing calibration)

APPENDIX F

G.4 Percent Recovery

$$\%R = \frac{Measured}{Expected} x100$$

where: %R = Percent recovery
Measured = Measured concentration of internal standard
Expected = Expected concentration of internal standard

G.5 Matrix spike percent recovery

$$Conc_{MS} = \frac{SSR - SR}{SA} x100$$

where: SSR = Spiked Sample Recovery
SR = Sample Result
SA = Spike Added

G.6 Relative Percent Difference

$$RPD = \frac{|R_1 - R_2|}{\frac{R_1 + R_2}{2}} x100$$

Where,

R1 = first sample value (original)
R2 = second sample value (duplicate)

G.7 Sample Quantitation Limit

$$SQL = CRQL_{sow} xDFx \frac{SOW\ Weight}{Sample\ Weight} x \frac{SOW\ Aliquot}{Sample\ Aliquot} x \frac{1}{\%S}$$

where: $CRQL_{sow}$ = contract required quantitation limit
DF = dilution factor
% S = percent solids (100 - % moisture)/100

APPENDIX F

Results

G.8 Waters:

$$\mu g/L = \frac{A_x x I_s x D_f}{A_{is} x RRF x V_o}$$

G.9 Soils (low level - dry weight basis):

$$\mu g/Kg = \frac{A_x x I_s}{A_{is} x RRF x W_s x D}$$

G.10 Soils (medium level - dry weight basis):

$$\mu g/Kg = \frac{A_x x I_s x V_t x 1000 x D_f}{A_{is} x RRF x W_s x V_a x D}$$

where:

A_x	=	area of the characteristic ion for compound being measured
A_{is}	=	area of characteristic ion for the internal standard
I_s	=	amount of internal standard added (ng)
RRF	=	daily response factor for compound being measured
V_o	=	volume of water purged (ml)
W_s	=	weight of sample (g)
D	=	percent solids
V_t	=	volume of methanol (This volume is typically 10.0 mL).
Df	=	dilution factor
V_a	=	volume of the aliquot of the methanol extract (uL) added to reagent water for purging

APPENDIX G

QC Acceptance Criteria[a]
Method 8270C - Semivolatiles

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L) (%)	Range p, p_s
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27-133
Benz(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
β-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl) ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl) ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26-155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152

APPENDIX G

Compound	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{x}$ (µg/L) (%)	Range p, p_s
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113
Ideno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in µg/L.

$\bar{x}$ = Average recovery for four recovery measurements, in µg/L.

p, p_s = Percent recovery measured.

D = Detected; result must be greater than zero.

a Criteria from 40 CFR Part 136 for Method 625.

Source: *USEPA Test Methods for Evaluating Solid Waste. SW-846* third edition, November 1986, Final Update III.

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED: Page 1 of ~~1~~ 2

Paducah Site-Wide
Procedure Type
(Company, Functional, Project, Subcontractor)

~~PA-5106: PCB and~~ Pesticide Data Verification & Validation
(Procedure Number and Title)

~~12/10/1998~~ 2-22-1999
(Procedure Date)

Rev. 0
(Rev. Number)

Reason for Changes: Transitioning BJC Procedures

SECTION	DESCRIPTION OF CHANGE
General	When reference is made to Lockheed Environmental Management and/or Bechtel Jacobs LLC personnel or contract, change the reference to Paducah Remediation Services (LLC) personnel or contract... Note that the procedure numbers will not change until after the first revision by PRS LLC.
General	Change Bechtel Jacobs LLC Environmental Management (EM) Projects to Paducah Remediation Services (LLC) Environmental Management (EM) Projects.
General	Attached is a cross walk from the Lockheed Environmental Management and the old Bechtel Jacobs LLC procedure numbers to the current Bechtel Jacobs LLC procedures. The procedure will be re-numbered with it is revised after transition.
Purpose	Delete the following sentence "This procedure applies to all PCDD/PCDF data verification and validation activities performed by or for the ER Program at the East Tennessee Technology Park (ETTP), Oak Ridge National Laboratory (ORNL), Y-12 Plant (Y-12), Paducah Gaseous Diffusion Plant (PGDP), and Portsmouth Gaseous Diffusion Plant (PORTS) sites, as well as those performed by or for ER's Off-Site Program." And replace it with the following "This procedure applies to all PCDD/PCDF data verification and validation activities performed by or for the ER Program at the Paducah Gaseous Diffusion Plant (PGDP) sites, as well as those performed by or for ER's Off-Site Program."

Source Documents	This procedure was last revised in 1999 and the source data lists Martin Marietta Energy Systems Inc. procedures. It is anticipated that PRS LLC will review and up date this procedure, and at that time update the references to match the existing work flow.

Initiator/Originator: Darryl Poteet — 4/7/06
(Print Name / Sign Name) — Date

REVIEWED BY: D McDaniel — 4/7/06
Discipline/Task Manager — Date

APPROVED BY: Diane McDaniel — 4/7/06
Project Manager — Date

BJC to PRS Position/Title/Organization Matrix
for PA-5106 PCB and Pesticide Data Verification and Validation

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
ER-DQP Validation Coordinator	→	Environmental Monitoring/Sample Mgr
Projec Manager	→	Project Manager
Data Verifier	→	Data Entry Specialist
Data Validator	→	Statistician/Validator
Data Validation Coordinator	→	Statistician/Validator
Analytical Services Coordinator	→	Contract Compliance Screener/lab Coordinator
Records Management Coordiantor	→	Data Entry Specialist
Data Management Coordinator	→	Data Manager/Network Specialist
	→	
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	→	

BECHTEL JACOBS
Bechtel Jacobs Company LLC

BJC-ES-01, Environmental Services Procedures Manual	ES-B-0811	REV. NO. 0
TITLE: PESTICIDE AND PCB DATA VERIFICATION AND VALIDATION	EFFECTIVE DATE: 02/22/99	Page 1 of 56
	PREPARER/POC: Beth Mitchell	
	APPROVED BY/DATE: R. B. Barber 12/10/98 (Signature on File)	

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REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release Replaces ERWM/ER-P2211	All

PURPOSE

This procedure defines the minimum requirements, responsibilities and methodology for the pesticide and PCB data verification and validation processes within the Bechtel Jacobs Company Environmental Restoration (ER) Program Organization. This procedure applies to all pesticide/PCB data verification and validation activities performed by or for the ER Program at the East Tennessee Technology Park (ETTP), Oak Ridge National Laboratory (ORNL), Y-12 Plant (Y-12), Paducah Gaseous Diffusion Plant (PGDP), and Portsmouth Gaseous Diffusion Plant (PORTS) sites, as well as those performed by or for ER's Off-Site Program.

This procedure provides requirements for developing and implementing a validation methodology for pesticide and PCB Contract Laboratory Program (CLP) and SW-846 (8081A and 8082) analytical methods primarily for analytes in aqueous and soil/sediment matrices. It is flexible enough to allow evaluation of data usability for project-specific Data Quality Objectives (DQO). Data produced by analytical methods for which this procedure provides limited guidance (i.e., Appendix A of 40 CFR Part 136 Protection of Environment, Appendix A - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, or "Superfund Low Concentration Statement of Work" methods) may necessitate development of modified criteria from this procedure; however, the general validation strategy outlined in this document should be applicable.

SCOPE

Specifications in this procedure should be incorporated into project documentation such as the Quality Assurance Project Plan, into contractual Statements of Work (SOW) between the project and the analytical laboratories, and into contractual validation SOWs between the project and the firm chosen to validate data. If validation is performed by individuals within the project, the SOW is not required but a mechanism to specify validation requirements is recommended. This procedure shall be used as a baseline to create project-specific checklists needed to perform pesticide/PCB data verification and validation. Guidance in developing and implementing a validation plan is provided in the procedure P2213, "Data Validation Plans for Environmental Restoration Projects". Examples of checklists for implementing this procedure are provided in Attachments B and C; current versions of these checklists are readily available from the ER Data Quality Program Forms Reference Library (423/241-4677) in either hardcopy or electronic format.

In this procedure, the words "shall" and "must" are used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission, neither a requirement nor a recommendation. In conformance to this procedure, all verification and validation shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

OTHER DOCUMENTS NEEDED

- *Data Quality Objectives Process for Superfund.* EPA/540/G-93/071, September 1993.
- *Guidance for the Data Quality Objectives Process.* EPA QA/G-4. August, 1994
- *USEPA Contract Laboratory Program Statement of Work for Organics Analysis.* March 1990, (Revision OLM03.1, August 1994).
- *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review.* EPA-540/R-94/012. February, 1994.
- *USEPA Test Methods for Evaluating Solid Waste.* SW-846 third edition, November 1986. (Final Update III, December 1996).

WHAT TO DO

A. General Information

1. Data Verification

Data verification is defined as a systematic process, performed by either the data generator (on-site or fixed base laboratory) or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

If verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

Data verification, at the project level, may be conducted either by the data verifier or by the data validator, whichever expedites the review process. If verification is conducted independently of the validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and statement of work, in terms of the required deliverables. The second activity entails various checks of the quality of the data, the purpose of which is to alert the validator that a non-correctable problem has occurred and that data may need qualification. If verification is conducted by the validator, the first activity is conducted similarly, but the second activity may result in prompt qualification of data.

Data verification should provide a mechanism for problem resolution with the laboratory; it should not be exclusively an after-the-fact identification of non-correctable deficiencies.

A verification report is written by the data verifier and takes, as input, the steps in this procedure that are listed as "Verification." The data verifier does not qualify data if verification criteria are not met but indicates in the verification report the circumstances surrounding correctable and non-correctable problems in the data package which will be transferred to the validator for possible qualification. The verification report must be in a standard format, and must remain associated with the analytical data package. A verification report which includes all verification checks in this procedure is provided in Attachment B.

2. Analytical Data Validation

Analytical data validation, including field and laboratory data review, is defined as a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data which may result in qualification of the data. Data validation is not performed by the analytical laboratory. Data validation provides a level of assurance, based on a technical evaluation, that an analyte is present or absent, and if present, the level of uncertainty associated with the measurement, and occurs prior to drawing a conclusion from a body of data. Analytical data validation includes a technical review of a laboratory data package covering the evaluation of quality control samples, the identification and quantitation of analytes, and the effect of deficiencies in quality on analytical sample data.

A validation report that includes the results of validation activities must be completed by the validator and takes, as input, the verification report and the steps in this procedure that are listed as "Validation." A validation report must be completed for every SDG that passes through validation. To expedite writing the validation report, comments must include explanations for qualification only if the reason codes do not adequately describe justification for qualification. Comments on data qualification for which reason codes adequately describe qualification reason are not necessary. A validation report which includes all validation checks in this procedure is provided in Attachment C.

Where professional judgement is used, a second opinion on choice of qualification of data should be sought. Consensus on professional judgement should be documented in the validation report.

B. Requirements

1. General Requirements

   a. This procedure will become effective thirty (30) days following the approval date to allow for distribution and initiation of training.

b. The actions described in this document must serve as the baseline for incorporation into project verification/validation activities. Project-specific verification/validation procedures applying to pesticide and PCB compounds and analytical methods not covered in this document must be submitted to the ER Data Quality Program for review and approval prior to use. Verification and validation activities subcontracted outside Bechtel Jacobs Company must adopt requirements consistent with those provided in this procedure.

2. Analytical Statement of Work Requirements

Implementation of this procedure is expedited through the agreement of work to be performed by an analytical laboratory in the form of a project-specific laboratory Statement of Work (SOW). Deliverable requirements specified in the analytical SOW must be consistent with the requirements of this procedure.

3. Validation Statement of Work Requirements

The validation SOW must be written consistent with the requirements and specifications of this procedure. The validation SOW is prepared by the ER project team and functions as the mechanism by which validation implementation requirements are communicated from the project team to the validation organization.

4. Validation Data Qualification Requirements

When qualification conditions are met, a qualifier and reason code(s) shall be physically and/or electronically associated with the affected result(s). If more than one reason code is applied, each should be separated by commas. The listing of available reason codes is presented in Attachment D. If data are not qualified, an equals sign ("=") is entered on the sample result. As directed in the validation SOW or as appropriate, reason codes may be applied without qualifiers.

5. Validation Strategy Worksheet Requirements

The worksheet presented in Attachment E must be completed by the project to identify the working pesticide and PCB data validation approach. For worksheet items where direct implementation of this procedure is not appropriate, an alternative approach must be provided and must be submitted to the ER-Data Quality Program (DQP) for approval. Establishment of the data validation strategy and plan is described in ERWM/ER-P2213), "Data Validation Plans for Environmental Restoration Projects".

6. Verification and Validation Reporting Requirements

The verification report (Attachment B) and validation report (Attachment C) must be completed for every SDG that passes through validation. Both the verification and validation reports must be completed regardless of who performs the reviews.

NOTE: The responsibilities outlined in this section do not preclude an individual assuming multiple responsibilities.

C. Responsibilities

ER-DQP Data Validation Coordinator — The ER-DQP Data Validation Coordinator is responsible for approval of alternative and non-routine verification and validation activities, and for providing training and technical support to ER projects in the areas of data verification and validation.

Project Manager — The Project Manager is responsible for oversight of the validation activities occurring within the project as a result of implementation of the requirements of this document.

Data Verifier — The Data Verifier is responsible for performing the verification actions listed in this procedure, including all items in Sect. VII listed as "Verification" and the writing of a verification report.

Data Validator — The Data Validator is responsible for performing the validation actions listed in this procedure, including all items in Sect. VII listed as "Validation" and the writing of the validation report.

Data Validation Coordinator — The Data Validation Coordinator is responsible for developing a data validation plan for the project, for incorporating the requirements of this procedure into the data validation plan for the project, for completing the validation implementation form (Appendix E), and for interfacing with the laboratory concerning data package deficiencies.

Analytical Services Coordinator — The Analytical Services Coordinator is responsible for interfacing with the data validation coordinator to ensure that analytical statements of work incorporate deliverables required by this procedure so that data packages from the laboratory will be appropriate for verification and validation.

Records Management Coordinator — The Records Management Coordinator is responsible for interfacing with the data validation coordinator in transferring data packages and verification/validation reports, and in providing a system of storage and control of these documents.

Data Management Coordinator

The Data Management Coordinator is responsible for interfacing with the data validation coordinator in transferring validated sample data and in entering validated data into the project database.

D. Action Steps – Pesticide/PCB Data Verification and Validation

1. Overall Implementation of Validation

NOTE: The following is a step-by-step approach to implement analytical data verification and validation activities for ER projects.

Data Validation Coordinator and Project Team

a. Validation Strategy and Plan Development

- Develop validation strategy based on inputs identified through the Data Quality Objectives (DQO) process according to ERWM/ER-P2213, "Data Validation Plans for Environmental Restoration Projects".
- Complete the Pesticide/PCB Data Validation implementation form (Attachment E).
- Write a validation Statement of Work to communicate verification and validation requirements to the organization performing that work for the ER project.

Analytical Services Coordinator

b. Analytical Statement of Work

- Write the analytical Statement of Work which communicates laboratory requirements from the ER project to the analytical laboratory.

Verifier and Validator

c. Implementing Verification and Validation

- Implement verification and validation by completing the forms (Attachments B and C, respectively).

NOTE: All laboratory contractual criteria apply to both chromatographic columns; however, results should be qualified based on quality considerations of the quantification column. If quality deficiencies occur on the confirmation column, do not qualify reported results. In instances of dilutions or sample reanalyses, the validator must indicate which data are of greatest usability from the respective column analysis.

2. Custody of Samples and Sample Documentation

The chain of custody (COC) is relevant to the representativeness of the sample to its origin. It is also legal documentation of sample possession. The COC documents the personal accountability of the samples, and any break in that signature series could result in jeopardizing the legal admissibility of the sample data.

While verification/validation cannot assure a sample has truly been in custody from the field to the final result, an evaluation of field and laboratory COCs will provide the best indicator. A sample is in custody if any of the following conditions are met:

- it is within one's possession;
- it is within one's view, after being in one's possession;
- it was in one's possession and then was secured to prevent tampering;
- it is placed in a designated secure area.

Verification includes CLP Form I header checks for accuracy from the COC. If header information is incorrect when compared to the COC, the verifier will mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, every attempt should be made to verify the true identity of each sample. Custody problems will affect the representativeness of the sample(s).

Data Verifier

a. Verification

- Trace the chain of signatures of all samples in the reporting batch from field sampling through receipt at the laboratory. If there is a break in the signature chain, indicate this problem in the verification report.
- Verify the headers of sample results summary reporting forms against COCs for accuracy. If header information is incorrect when compared to the COC, mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, make every attempt to verify the true identity of each sample; however, if sample representativeness remains in doubt, note the occurrence on the verification report.

Data Validator

b. Validation

- Qualify sample data which is not traceable through signature records on COCs "R."

3. Holding Times, Turn-around Times, and Sample Preservation

NOTE: Holding Times have been established by EPA to provide a maximum period of time in which an environmental sample will remain representative of its sampling location. Holding time begins when a sample is collected in the field, rather than date of sample receipt, and ends with extraction and analysis. If the final reported data results from a dilution or reanalysis of the sample, this analysis must have been completed within the holding time.

a. Deliverables

- Field chains of custody
- Laboratory chains of custody
- Dates of collection, preparation and analysis for all samples

b. Criteria

The following table provides criteria for holding times:

Holding Time Criteria

Matrix	Container		Preservative		Holding Time	
	OLM03.1	SW-846	OLM03.1	SW-846	OLM03.1	SW-846
Water samples	amber glass with Teflon liner	amber glass with Teflon liner	4 ± 2°C	4 ± 2°C	7 days * 40 days **	7 days * 40 days **
Soil or sediment samples	Glass, or closed end tubes (e.g. brass sleeves)	glass with Teflon liner	4 ± 2°C	4 ± 2°C	14 days * 40 days **	14 days * 40 days **

* Time from collection of sample to extraction.
** Time from extraction of sample to analysis.

Data Verifier

c. Verification

- Verify the presence of the pertinent chains of custody.

- Turn-around time is evaluated as strictly a contractual issue. An agreement should be established between the client and the laboratory concerning turn-around times for reported data. If turn-around times are not satisfied, a contractual mechanism should provide for appropriate action.

Data Validator

d. Validation

- Qualify detected compounds "J" and compounds ≤ Contract Required Quantitation Limit (CRQL) "UJ" if holding times are exceeded. Data may be qualified "R" if the reviewer determines that the effect of holding time exceedence on sample results were significant; however, organochlorine compounds need not be rejected in soil samples for exceedence of holding times due to relative stability in this matrix.

- As no validation criteria has been established for soil samples, it may be appropriate to apply water holding time criteria to soils; however, if that criteria are applied, justification must be provided in the validation report.

- If samples have exceeded temperature requirements, and sample results seem to be affected, qualify detected compounds "J" and compounds ≤ CRQL "UJ." Data may be qualified "R" if the reviewer determines the effect of temperature exceedence has had a significant effect on the sample results.

4. GC/ECD Instrument Performance Check (Required for CLP method)

The instrument performance check uses standard materials to ensure adequate resolution and instrument sensitivity. The GC/ECD Performance Check consists of the Resolution Check Mixture and Performance Evaluation Mixture (PEM).

a. Deliverables

- CLP Form VI PEST-4,5; CLP Form VII PEST-1; CLP Form VIII PEST

- Raw data (required for confirmation).

b. Frequency

- The Resolution Check Mixture, which contains the 9 pesticide and surrogate compounds that are most difficult to resolve, must be analyzed at the beginning of every initial calibration sequence on each GC column for each instrument used for analysis.

- The Performance Evaluation Mixture (PEM), which contains 8 pesticides and surrogates, must be analyzed after the resolution check mixture at the beginning of the initial calibration sequence. The PEM must also be analyzed at the beginning of every other 12-hour analytical period.

c. Performance Criteria

- For the Resolution Check Mixture, resolution on both GC columns must be ≥ 60%.

- For PEM, resolution must be ≥ 90% for all peaks on both columns.

- Retention times (RT) must be within retention time windows, centered around the mean retention times determined from the three-point initial calibration using Individual Standard mixtures.

- Relative Percent Difference (RPD) between calculated amount and true amount for single component pesticides and surrogates must be within ±25%.

- Percent breakdown for 4,4'-DDT and Endrin in each PEM must be ≤ 20%. Combined percent breakdown must be ≤ 30%.

Data Verifier

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason code "C07" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "C06" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

- If the resolution is < 60 % on either column for Resolution Check Mixture, qualify detected target compounds "J" and nondetects with retention times in the region of coelution "R" as appropriate.

- If resolution is < 90% between any peaks for PEM, qualify affected detected target compounds "J" and affected quantitation limits "R" as appropriate.

- If the retention times of PEM compounds do not fall within the retention time windows, the associated sample results should be carefully evaluated. If no peaks in the sample chromatograms are close to the expected retention time window of the compound of interest, no action is usually necessary.

- If peaks occur in the sample chromatograms that are > CRQL and close to or within expected retention time window of the compound of interest, affected detected compounds may be qualified "NJ" and non-detected compounds may be qualified "UJ." Additional effort may be necessary to determine if sample peaks represent the target compounds.

NOTE: (1) Inspect to determine if 3 or more standards containing the pesticide of interest were run within a 72 hour period that the sample(s) was run. (2) If 3 or more such standards are present at this frequency, the mean and standard deviation of the RT window can be determined. (3) If all standards and matrix spikes fall within this revised window, valid detects and non-detected sample results can be determined using this window. (4) If the validator cannot do anything with the data to resolve the RT window problem of concern, all detects and quantitation limits should be qualified "R."

- If the Relative Percent Difference (RPD) between the calculated and true amounts for each single component pesticides and surrogates is outside the control limits, ±25%, qualify all affected detected target compounds "J" and all affected quantitation limits as "UJ."

- The following provides qualification action if Percent Breakdown of 4,4'-DDT or Endrin is > 20% or the combined Percent Recovery is > 30%.

  — Detected compounds DDT or Endrin should be qualified "J." DDD, DDE or Endrin aldehyde and Endrin ketone, if detected, should be qualified "NJ."

  — DDT and Endrin should be qualified "R" if not detected, and if DDD and DDE or Endrin aldehyde and Endrin ketone are detected.

5. Initial Calibration

Calibration is established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for pesticide and Aroclor compounds on the Target Compound List (TCL). Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run and of producing a linear calibration curve.

a. Deliverables

- CLP Form VI-PEST 1,2,3; VII-PEST 2; VIII

- Raw data (required for confirmation).

b. Frequency

- Initial Calibration must be performed before any samples are analyzed on the GC/ECD or GC/ELCD system.

c. Performance Criteria

- For CLP single component compounds, 3 standards are used with the low point near the CRDL, the midpoint 4X the low, and the high point at least 16X the low, although the high point may be greater than this level. For SW-846, method 8081A, 5 standards are used for single component pesticides, with the lowest point near but greater than the MDL. The analysis of the multi-component pesticides employs a single point calibration, unless otherwise specified by the project.

- For CLP multi-component pesticide compounds and PCBs, one standard is required. For SW-846 method 8082, a minimum of five calibration standards containing a mixture of Aroclors 1016 and 1260 is required. Single standards of each of the other five Aroclors at a concentration near the midpoint of the calibration range, is required. A minimum of five calibration standards is required when analyzing individual PCB congeners.

- Individual Standard Mix A and B (INDA and INDB)The following criteria apply to Individual Standard Mixtures A and B (INDA, INDB):

  — Resolution between any adjacent peaks in the midpoint concentration must be ≥ 90%.

  — At least one chromatogram from each Individual Standard Mixture must yield peaks with recorder deflections between 50-100% of full scale.

  — Percent Relative Standard Deviation (RSD) of Calibration Factors (CF) for single component pesticides must be ≤ 20%; except, up to two single component pesticides per column may exceed 20 but must be ≤ 30%. For surrogates, % RSD must be ≤ 30%.

  — For SW-846 methods, if results are quantitated directly from the calibration curve, the correlation coefficient from the curve must be ≥ 0.99.

  — Cfs may be calculated based on peak height or area, but the choice must be consistent.

- SW-8082 PCB Analysis

  — When determining PCB congeners by the internal standard procedure, the percent RSD of response factors must be ≤20%.

  — When determining PCBs or Aroclors by the external standard technique, the percent RSD of calibration factors must be ≤20%.

- Absolute retention times for single-component pesticides must fall within the appropriate retention windows. Absolute retention times are determined from the three point initial calibration using INDA and INDB.

— Alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, Heptachlor, Aldrin ±0.05 minutes

— Alpha-Chlordane, gamma-Chlordane, Heptachlor epoxide, Dieldrin, Endrin, Endrin aldehyde, Endrin ketone, DDD, DDE, DDT, Endosulfan I, Endosulfan II, Endosulfan sulfate, Methoxychlor, Aroclors, Toxaphene ±0.07 minutes

— Tetrachloro-m-xylene ±0.05 minutes

— Decachlorobiphenyl ±0.10 minutes

- Retention time for multi-component compounds is determined for 3 to 5 peaks for each compound. The retention time acceptance window is set at ± 0.07 minutes from the retention time of the respective 3 to 5 chosen peaks.

Data Verifier

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms are not included, and/or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and/or the required standard levels were not used, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason code "C07" on the affected data if non-correctable deliverable deficiencies have occurred, and place the following reason codes if the following conditions are met, qualify only if the deviation indicates an adverse effect on data quality.

  — Initial calibration sequence was not followed. "C03"

  — Appropriate number of standards were not used or "C24"

  — Inappropriate concentrations. "C18"

- If resolution between adjacent peaks in the midpoint concentration for INDA and INDB are < 90%, qualify positive results "J" and non-detected target compounds with retention times in the area of coelution "R" as appropriate.

- If the sample concentration is outside the calibration curve and it was not diluted and reanalyzed, qualify all positive results as "J."

- If %RSD of calibration factors is > 20% for more than two single component compounds, > 30% for the two single component compounds that may exceed 20% or > 30% for surrogates, qualify all positive affected results "J" and non-detect sample quantitation limits "UJ."

- If %RSD of calibration or response factors is >20% for multicomponent compounds, quality all positive affected results "J" and non-detect sample quantitation limits "UJ".

- Raw Data Confirmation

  — Recorder deflections for peaks from at least one chromatogram must be >50% of full scale. If this criterion is not satisfied, qualify samples only if the peak heights or areas are small enough to preclude accurate quantitation of detected compounds.

6. Continuing Calibration

Continuing calibration ensures that the instrument(s) is capable of consistently producing acceptable qualitative and quantitative data. The instrument(s) is checked over specific time periods during the sample analysis.

a. Deliverables

- CLP Form VI-PEST 6,7; VII-PEST 1,2; VIII-PEST

b. Frequency

- Immediately following initial calibration, calibration verification for CLP methods must begin with the analysis of an instrument blank and a PEM. Throughout the analytical run, an instrument blank and a PEM must bracket one end of a 12-hour period while a second instrument blank and the midpoint concentration of INDA and INDB bracket the other end. For SW-846 methods, a calibration standard must be analyzed after each group of 20 samples (after every 10 is recommended).

c. Performance Criteria

- Resolution criteria

  — Resolution between any two adjacent peaks in the midpoint concentration in INDA and INDB must be greater than or equal to 90.0%.

- Retention time windows

  — The absolute retention time for each single component pesticide and surrogate in the midpoint concentration of INDA and INDB must be within the retention windows determined from the initial calibration. All samples injected after the last in-control standard are potentially affected.

- RPD Limits

  — For CLP methods, RPD between the calculated amount and true amount for each of the single component pesticides and surrogates in the midpoint concentration in INDA and INDB must not exceed ±25%. For SW-8081A, response factors for continuing calibration must be within ±15% of the initial calibration.

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms, frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason "CO7" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "CO6" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

- Resolution criteria

  — If resolution criteria are not satisfied, qualify detected compounds "J" and nondetects "R" as appropriate.

- RPD Limits

If RPD between the calculated amount and true amount for each of the single component pesticides and surrogates in the midpoint concentration in INDA and INDB, or continuing calibration standard, is > 25%, qualify affected detected target compounds "J," and nondetects "UJ."

7. Blanks

Blank analyses serve to determine the existence and magnitude of contamination resulting from laboratory or field activities. If contamination is found in any blank, all associated data must be carefully evaluated to determine whether or not there is a systemic problem effecting greater than one sample or if the contamination is an isolated occurrence.

a. Deliverables

The minimum deliverables necessary to complete the checks are described in this section:

- Method Blank: CLP Form I, or equivalent for SW-846 methods, for each method blank.
- Instrument Blank CLP Form IV-PEST and CLP Form VIII-PEST
- Raw data for each blank (required for confirmation).

b. Frequency

- Method blanks must be extracted for each 20 samples of similar matrix in each SDG or whenever a sample extraction procedure is performed.
- For CLP methods, an acceptable instrument blank must be analyzed at least once in each 12 hour period immediately prior to the analysis of either the PEM or INDA and INDB depending on the position in the analysis sequence. An instrument blank must be analyzed immediately after a sample containing compound(s) at high concentration(s) for carryover.
- The Sulfur Cleanup Blank (SCB) must be analyzed whenever part of a set of samples extracted together requires sulfur cleanup. If the entire set of samples associated with a method blank is subjected to sulfur cleanup, the method blank will suffice as a SCB, and no

separate SCB is required.

c. Performance Criteria

- Compounds detected in blanks analyzed under CLP must be at levels than less than the CRQL. Method blank performance criteria is not specified for SW-846 methods. For the purposes of validation, sample results shall be evaluated against CLP guidelines.

Data Verifier

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason code "B07" on the affected data if non-correctable deliverable deficiencies have occurred; qualify only if the deviation indicates an adverse effect on data quality.

- All blanks associated with the case must be evaluated against the sample results in the case. However, qualification should only be applied to those samples directly related to the affected blank (if more than one method blank is used per case). Blank CLP Form 4(s) are <u>not</u> qualified due to contamination.

- Any compound that is reported in both blank and sample must be evaluated; however, if the same compound is reported in sample(s) and more than one blank, the sample should be evaluated against the blank with the highest concentration of the compound. Sample results must <u>not</u> be modified by subtracting blank values.

- If sample concentration > CRQL and > 5 X Blank concentration, no qualification of result is necessary

- If sample concentration is > CRQL and < 5 X Blank concentration, qualify the reported result with "U"

- If sample concentration < CRQL and < 5 X Blank concentration, report SQL and qualify the SQL with "U"

- If gross contamination (saturated peaks in blank) is present

qualify, all affected results "R"

- If an instrument blank is not analyzed immediately after a sample showing compound(s) at high concentration(s), the validator must evaluate the analyses following the saturated sample analysis for carryover. Qualify reported compounds significantly affected by instrument carryover "J" or "R."

- Raw data confirmation

— Verify that results for the method blank are reported accurately on the CLP Form 1(s) and 4C(s) from the raw data.

8. Surrogate Standards

Surrogate spike recovery provides for establishing the performance of the laboratory extraction and analysis.

a. Deliverables

- CLP Form II PEST-1 and 2 (2E, 2F) or equivalent for SW-846 methods

- Raw Data (required for confirmation)

b. Frequency

- All samples, standard mixtures, PEMs, blanks, and matrix spikes are fortified with the two surrogates tetrachloro-m-xylene and decachlorobiphenyl (DCB).

c. Performance Criteria

NOTE: Performance criteria are not specified for SW-846 methods 8081A and 8082 for surrogate standard recovery, retention time, or RPD. For the purposes of validation, sample results shall be evaluated against CLP guidelines.

- Percent Recovery Limits

— The advisory percent recovery limits for the surrogates are 30-150% for both water and soil matrices. Consideration must be given to the fraction to which the surrogate is applied. For CLP analysis, the pesticide fraction should be compared to Tetrachloro-m-xylene, and the PCB fraction should be compared to Decachlorobiphenyl. For method SW-8081A, the surrogates DCB and tetrachloro-m-xylene are recommended. For SW-8082, DCB is used when PCBs are determined as aroclors, and tetrachloro-m-xylene is used for PCB congener analysis.

- Retention Time Windows

— The surrogates must fall within the retention time windows from the continuing calibration (tetrachloro-m-xylene ± 0.05 minutes; decachlorobiphenyl ± 0.10 minutes).

d. Verification

Data Verifier

- Verify the presence of the pertinent reporting forms, the identity of the proper surrogates and surrogate concentrations; if the required reporting forms or required surrogates or concentrations are not used, and these instances are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason code "S06" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "S05" if inappropriate surrogate concentrations have been used; and place reason code "S04" if inappropriate surrogate standards have been used; qualify only if the deviation indicates an adverse effect on data quality.

- Surrogate Recovery

— If surrogate recovery falls in the range of 10-30%, qualify detected compounds "J," and nondetects "UJ."

— If surrogate recovery falls in the range of 0-10%, qualify detected compounds "J," and nondetects "R," unless dilution is a factor in the percent recovery.

— If surrogate recovery is 0%, ensure that the surrogate peaks have been identified correctly. If the peak is truly not recovered, qualify detected compounds and nondetects "R," only if surrogate is not detected outside the retention window. If dilution is a factor, data may not need to be qualified. Indicate on the laboratory reporting forms which results are the most reliable.

— If surrogate recovery is > 150%, qualify detects "J;" nondetects do not require qualification.

- Retention Time Windows

— If surrogates are detected outside the above retention windows, the possibility of false negatives exists. It may be appropriate to qualify reported detection limits indicative of false negatives "R."

- Raw Data Confirmation

— Recalculate one surrogate recovery from raw data. Equation G6 in Attachment G is used for calculating surrogate percent recovery.

9. Matrix Spikes / Matrix Spike Duplicates (MS/MSD)

MS/MSD data are generated to determine long-term precision and accuracy of the analytical method on various matrices. Qualification must not be applied to sample data based on MS/MSD data alone, but should be used in conjunction with other QC parameters in judging data usability.

a. Deliverables

- CLP Form III PEST-1, 2 (3E, 3F)
- Raw Data (required for confirmation)

b. Frequency

- MS/MSD must be analyzed at a frequency of at least one MS/MSD pair per 20 field samples of similar matrix.

c. Performance Criteria

- Percent Recovery and RPD

- Inspect the percent recovery of the MS/MSD and ensure that the recoveries and RPD are within the following advisory limits:

MS/MSD Accuracy and Precision Criteria

	CLP SOW OLM03.1			
Compound	% R Water	RPD Water	% R Soil	RPD Soil
gamma-BHC (Lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4'-DDT	38-127	27	23-134	50

Data Verifier

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason code "M05" on the affected data if non-correctable deliverable deficiencies have occurred, and place reason code "M04" if frequency requirements have not been satisfied; qualify only if the deviation indicates an adverse effect on data quality.

- A determination shall be made concerning what extent that non-compliant MS/MSD data has on other sample data in regard the MS/MSD sample itself as well as specific compounds in samples associated with the MS/MSD. In those instances where it can be determined that the results of the MS/MSD affect only the sample spiked, then qualification should be limited to that sample alone. However, it may be determined that the laboratory is having a systematic problem in the analysis of one or more compound, which affects all associated samples. Positive results of non-spiked compounds may be qualified "J" as appropriate.

- Raw Data Confirmation

  — Recalculate one MS recovery from raw data. Equation G1 in Attachment G is used to calculate MS percent recovery.

10. QC Check Standard (SW-846 Methods only)

The QC Check Standard is not required for the CLP 3/90 SOW but is required for SW-846 methods 8081 and 8082. It is stressed that this standard must be the same matrix as the analytical samples (especially for aqueous samples) and prepared and analyzed to demonstrate initial proficiency of the method. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. The QC Check Standard is prepared from addition of a QC Check Standard concentrate into the appropriate clean matrix, extracted and analyzed.

a. Deliverables

- Report summary of all analytes in the QC Check Standard
- Raw data for each QC Check Standard (required for confirmation).

b. Frequency

- The QC Check Standard should be prepared and analyzed to demonstrate initial proficiency for the method, and must be repeated when new staff are trained, or significant changes in instrumentation are made.

c. Performance Criteria

- The QC Check Standard must be analyzed and must fall within limits specified by the determinative method. If no limits are specified, recovery limits of 70-130% should be used in evaluating results.

Data Verifier

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all instruments used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the

verification report.

e. Validation

- Place reason code "K04" on the affected data if non-correctable deliverable deficiencies have occurred; qualify only if the deviation indicates an adverse effect on data quality.

- If the QC Check Standard was prepared and analyzed and percent recovery criteria are not satisfied, qualify those compounds in the affected samples "J," using professional judgement to qualify "R."

- If the QC Check Standard was analyzed only (i.e. not prepared) it will provide limited value for method accuracy. Qualification should be applied only if the QC Check Standard and other QC data within the batch indicate that the accuracy of reported compounds has been affected.

- If the percent recoveries of the QC Check Standard compounds are high when compared with laboratory-generated limits, qualify detects "J." If the percent recoveries of the QC Check Standard compounds are low when compared with laboratory-generated limits, qualify detects "J," and nondetects "UJ" or "R;" however, other QC in the SDG must be evaluated prior to rejecting data based on QC Check Sample recovery.

11. Pesticide Cleanup Check

Pesticide cleanup is performed to remove matrix interferences from sample extracts prior to analysis. Florisil cleanup significantly reduces these interferences caused by polar compounds. Gel permeation chromatography (GPC) is used to remove high molecular weight contaminants that can interfere with the analysis of target compounds.

The pesticide cleanup procedures are checked by fortifying the cleanup columns and cartridges, and verifying the percent recovery of pesticides which may be affected through the cleanup procedure.

a. Deliverables

- CLP Form IX (9A and 9B)

- Raw data (required for confirmation)

b. Frequency

- For CLP Methods, Florisil cleanup is used on all sample extracts, and GPC is used for the cleanup of all soil extracts and for water sample extracts containing high molecular weight components that interfere with the analysis of the target compounds.

- SW-846 Method 3600A requires that the cleanup procedure be performed on all samples and quality control samples in order to evaluate that the cleanup procedure is functioning adequately. GPC is performed at the discretion of the analyst.

c. Performance Criteria

- For CLP Methods, each lot number of Florisil must be checked by spiking with 2,4,5-trichlorophenol and the midpoint concentration of INDA. Florisil cartridges are considered acceptable for use if recoveries of 80-120% in INDA are reported and if percent recovery of 2,4,5-trichlorophenol is < 5%, and if no peaks interfering with target compounds are detected.

- The calibration of the GPC unit must be checked at least once each 7 days by fortifying with two check mixtures: the MS solution and a solution of 0.2 µg/ml of Aroclors 1016 and 1260. Calibration is acceptable if percent recovery of the MS solution are within 80-110%, and the Aroclor pattern matches patterns of those compounds previously analyzed. A GPC blank must be analyzed after each GPC calibration and is acceptable if the GPC blank does not exceed one-half the CRQL for any target compounds.

- For the SW-846 method, if quality control samples show percent recoveries of surrogates that are outside criteria, evaluate whether the recovery may have been due to matrix effects or loss through extraction prior to qualification.

Data Verifier

d. Verification

- Verify the presence of the pertinent reporting forms. If the required reporting forms or frequency of analysis is not satisfied for all cleanups used for sample analyses in the SDG, and these occurrences are considered non-correctable problems, indicate this in the verification report. As this is a contract compliance issue, the occurrence should be communicated to the validator in the verification report.

e. Validation

- Place reason code "V04" on the affected data if non-correctable deliverable deficiencies have occurred; qualify only if the deviation indicates an adverse effect on data quality.

- If frequency requirements have not been satisfied, sample data should be qualified only if the deviation indicates an adverse effect on data quality.

- If percent recovery of surrogates is <10%, the validator should qualify non-detects "R." If percent recovery of surrogate or pesticide compounds is > 120%, professional judgement must be used in qualification of detected target compounds; non-detected compounds need no qualification.

- Raw Data Confirmation

  — If criteria are not satisfied, raw data should be examined for the presence of high molecular weight contaminants. If these contaminants are present and significantly preclude accurate quantitation of target compounds, affected detected compounds may be qualified "J" and nondetects qualified "R."

12. Target Compound Identification

a. Deliverables

- CLP Form I, or equivalent for SW-846 methods

- Raw Data (required)

b. Performance Criteria

- Retention times of both of the surrogates, matrix spikes, and reported compounds in each sample must be within the calculated retention time windows on both columns. Surrogates, MS/MSD, and reported compounds in each sample must elute within the calculated retention time windows on <u>both</u> columns. Tetrachloro-m-xylene must elute within ± 0.05 minutes of the mean retention time determined from initial calibration; and decachlorobiphenyl (DCB) must elute within ± 0.10 minutes of the mean retention time determined from initial calibration.

c. Validation with Raw Data Confirmation

- Ensure that all samples have been analyzed on the two contract-required chromatographic columns. If samples have not been analyzed on the two columns, place reason code "Q05" on detects and nondetects.

- Check sample chromatogram for peaks close to the expected retention window of the pesticide of interest. If no peaks are present either within or close to the retention window, non-detected values can be considered valid.

- If the affected sample chromatogram contains peaks > CRQL, and either close to or within the expected retention window of the pesticide of interest, the possibility of false negatives exists. Calibration retention time windows should be consulted to determine appropriate window of elution; and the sample chromatogram should be inspected for occurrences of high concentration compounds or contaminants, or matrix interferences that may affect the retention time window of the sample.

- Ensure that an instrument blank was be analyzed immediately after a sample containing compound(s) at high concentration(s) for carryover.

- If retention time criteria are not met, the possibility of false positives and false negatives exist. All target compounds reported as detected should be changed to non-detected status with the SQL reported instead of a detected value; use the following guidance when reporting SQL(s) instead of detected values:

- If a misidentified peak was sufficiently outside the target pesticide retention time window, the reported values may be false positive and should be replaced with the SQL value.

- If a misidentified peak poses an interference with potential detection of a target peak, then the reported value should be considered and qualified "R."

- If multi-component target compounds exhibit marginal pattern-matching quality, professional judgement should be used to establish whether the differences are due to environmental "weathering" (degradation of the earlier eluting peaks relative to the later eluting peaks). If the presence of a multi-component pesticide is strongly suggested, results should be reported "NJ."

- GC/MS Confirmation

  — The validator must verify that GC/MS confirmation was run on samples if concentrations of any compound exceed 10 ng/uL. If the samples have been analyzed for the presence of volatiles or semivolatiles, a cross-reference should be made to the TICs in these fractions to determine the possible identity of artifacts detected in the pesticide/PCB fraction.

- Pattern Matching of Multi-Component Compounds

  — If multi-component pesticides/PCBs exhibit marginal pattern-matching quality, professional judgement should be used to establish whether the differences are due to degradation of earlier eluting peaks relative to later eluting peaks. If the presence of a multi-component pesticide is strongly suggested, results should be reported "N." If a pattern matches more than one Aroclor, the best match should be chosen.

- Reported results may be recalculated from raw data using equations G8, G9, and G10 in Attachment G.

13. Manual Recalculation of Analytical Results

The accuracy and consistency of sample result calculation by the laboratory can be addressed two different techniques. The application of each strategy depends on the laboratory's ability to minimize transcription during reporting, and how familiar the project is with the performance of the laboratory. If sample results are produced primarily through software processing and minimal transcription is performed in the laboratory, the data system(s) can be evaluated during an audit or surveillance by performing two different tests on the software (1) supply the data system a consistent set of input designed to provide a consistent set of output, and (2) supply the data system a set of nonconforming data to test the error detection routines. An additional evaluation of the laboratory's software configuration control and security is also necessary. Through this technique, a high level of confidence can be gained in the laboratory's reporting techniques and will result in a minimal need for manual recalculation of sample results.

If the laboratory has a high rate of manual transcription in generation of sample results, the project may choose to manually recalculate sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contacting the laboratory may be necessary to resolve the problem. Data may be qualified "R" as a last resort if no actions can reproduce reported values.

Calculations for compound quantitation and rounding rules can be found in

Attachment G.

14. CRQLs and SQLs

CRQL(s) have been developed to enable the laboratory to meet realistic detection limit goals.

Due to deviations from method-specified sample weights, extract volume or aliquot used in analysis, or due to dilution or soil percent moisture, CRQLs are modified accordingly and are termed "sample quantitation limits" (SQL).

a. Deliverables

- CLP Form I or equivalent for SW-846 analytical methods for all samples

b. Frequency

- CRQLs or SQLs are reported for all compounds that are not detected above the method MDL.

Data Verifier

c. Verification

- For all samples, the SQL must be less than the Required Detection Limit (RDL), which are identified and communicated to the laboratory in the laboratory SOW. If the SQL is greater than the RDL, this may indicate matrix-related problems or analytical conditions precluding RDL achievement.

- All sample results shall be reviewed to determine RDL compliance. In cases where the SQL is greater than the RDL, the project may decide to request a reanalysis.

- Verify that compounds detected at levels below the SQL have been qualified "J" by the laboratory.

d. Validation

- For one non-detected compound in each sample and blank, verify that CRQL(s) have been adjusted, if necessary. No additional validation qualifiers are necessary for results detected below the SQL unless directed in other sections of this document.

- Quantitation limits effected by large off-scale peaks should be qualified "R."

- Single peak pesticide results are checked for agreement between the two column analyses. The potential for co-elution should be considered if a sufficiently larger concentration of a compound occurs on one column. If an interfering compound is indicated, an appropriate reporting strategy should be considered, or if the reported compound is significantly affected, the affected compound may be qualified "J" or "R."

- Equation G3 for modifications to the CRQL can be found in Attachment G.

RECORDS

Records arising from the actions in this document are as follows:

NOTE: These records become part of the analytical data package and must be controlled through a records management system.

- The verification report.
- The validation report.
- Copies of qualified, or unqualified results reports.
- Quality Assurance Records

SOURCE DOCUMENTS

- "Comprehensive Data Validation," ER/C-S2201, Rev. 0, Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee, October 29, 1993.

- "Data Validation Plans for Environmental Restoration Projects," ERWM/ER P2213, Lockheed Martin Energy Systems, Inc., Oak Ridge, Tennessee.

- Data Management Plan for the Environmental Restoration Program, ES/ER/TM-88.

Attachment A
DEFINITIONS/ACRONYMS

Qualifier definitions and reason codes are listed in Appendix D.

Affected Sample Result: An affected sample result is a sample result considered to be affected significantly by a quality deficiency and is qualified accordingly through analytical data validation.

Analytical Data Validation: Analytical data validation is a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Analytical Data Verification: Analytical data verification is a systematic process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract which is performed by either the data generator or by an entity external to the data generator.
Batch: A batch is a group of samples prepared at the same time in the same location using the same method, not to exceed 20 samples of similar matrix.

Case: A finite, usually predetermined number of samples collected over a given time period from a particular site. A case consists of one or more sample delivery groups (SDG).

Chain of Custody (COC): The COC is the history of the transfer of samples from the time of sample acquisition through archival and disposal of samples.

Contract Required Quantitation Limit (CRQL): The CRQL is the minimum level of detection acceptable under the current CLP contract.

Correctable Problem: Correctable problems are deficiencies within data packages which may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation. Correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory. Correctable problems revealed during validation are those deficiencies with analyses that can be solved by either a second preparation and/or analysis of a sample.

Laboratory Duplicate: The laboratory duplicate is a randomly chosen split of a field sample into two aliquots prior to sample preparation. The purpose of a laboratory duplicate is to monitor the precision of the analytical method.

Matrix Spike: The matrix spike is a split of a field sample in which one half of the split is spiked with a known amount of analyte of interest prior to sample preparation. The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation.

Attachment A
DEFINITIONS/ACRONYMS
Page 2 of 3

Method Blank: The method blank is a laboratory-generated sample of the same matrix as the analytical samples, but in absence of the analyte of interest. The purpose of a method blank is to monitor the presence of contamination of the analyte of interest in the sample preparation and analysis processes.

Non-Correctable Problem: Non-correctable problems are deficiencies within data package which preclude the evaluation of data quality by predefined criteria. Non-correctable problems may be revealed during both data verification and data validation.

Quality-indicator Sample: Quality-indicator samples are those samples made ready in the laboratory which provide direct or indirect evaluation of the status of the analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

Relative Percent Difference (RPD): RPD is the measure of precision between two values, defined as the absolute value of the difference between two values divided by the mean of the two values.

Relative Response Factor (RRF): RRF represents the response of a compound to an analytical instrument relative to the response of an associated standard.

Relative Standard Deviation (RSD): RSD is the measure of precision between multiple values, defined as the standard deviation of multiple values divided by the mean of the values.

Required Detection Limit (RDL): The RDL is a contractually-specified detection limit which, under typical analytical circumstances, should be achievable.

Standard Reference Material (SRM): A material or substance of which one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Sample Delivery Group (SDG): A SDG is defined by one of the following, whichever occurs first:

- Case of field samples
- Each 20 field samples within a case

Each 14-day calendar period during which field samples in a case are received, beginning with receipt of the first sample in the SDG.

Attachment A
DEFINITIONS/ACRONYMS
Page 3 of 3

Sample Quantitation Limit (SQL): SQLs are detection limits based on CRQLs, which have been modified due to deviations from analytical method specifications such as sample weight and extract volume or due to dilution or percent moisture.

Sample Result: A sample result, as described in this procedure, is a numeric denotation of the concentration, amount or activity of a specific analytical parameter uniquely associated with an aliquot of environmental media.

Standard Reference Material (SRM): A SRM is a material or substance of which one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Surrogate: Non-target standard compounds added to every blank, sample matrix spike, matrix spike duplicate and standard; used to evaluate analytical efficiency by measuring percent recovery.
Technical Holding Time: For Pesticide and PCB, the time, expressed in calendar days, from sample collection to sample extraction and from sample extraction to sample analysis.

Traceable Reference Material (TRM): A TRM is a NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).

Turn-around Time: Turn-around time is contractually-specified as the length of time which elapses between laboratory receipt of the field samples and subsequent data receipt by the client.

Validation Statement of Work: The validation SOW is a document prepared by the ER project to function as the mechanism by which validation implementation requirements are communicated from the project to the validation organization.

Well Characterized Reference Material (WCRM): The WCRM may be derived from a field sample which has been well characterized through multiple analyses, providing a high level of confidence of the concentration in the sample. The WCRM may be submitted to NIST for characterization and classification as a Traceable Reference Material (TRM).

Attachment B
DATA VERIFICATION REPORT
Page 1 of 2

Pesticide and PCB Data Verification Report

Sample Delivery Group #:

Matrix:

Laboratory:

Laboratory SOW#:

This checklist is to be used for verification of Pesticide/PCB data packages, consistent with the procedure ES-B-0811. No qualifiers are placed through the process of verification.

Verified by:_____ Date:

Attachment B
DATA VERIFICATION REPORT
Page 2 of 2

	Y	N
Custody of Samples		
1. Are samples traceable through inspection of signature records on field and laboratory chains of custody?	___	
Action: For any samples not traceable upon inspection of chains of custody, indicate the identities below and where the break in traceability occurred.		
GC/ECD Instrument Performance Check		
2. Are Form VI PEST-4,5; Form VII PEST-1; Form VIII PEST or equivalent for SW-846 methods included in the data package?	___	
3. Has the performance check been run at the specified frequency?	___	
Initial and Continuing Calibration		
4. Are Form VI-PEST 1,2,3; VII-PEST 2; VIII for initial calibration and Form VI-PEST 6,7; VII-PEST 1,2; VIII-PEST for continuing calibration, or equivalent for SW-846 methods, included in the data package?	___	
5. Has initial calibration been performed prior to analysis of analytical samples?	___	
6. Has the appropriate sequence been followed for initial and continuing calibrations?	___	
7. Does an instrument blank and PEM bracket one end of a 12-hour period while a second instrument blank and midpoint INDA and INDB bracket the other end?	___	
8. Have the contract-required standard concentrations been used (or those specified in SW-846)?	___	
Method Blank		
9. Are Form I's, IV and VIII, or equivalent for SW-846 methods, included in the data package?	___	
10. Have frequency requirements for the method blank been satisfied?	___	
Surrogate Standards		
11 Are Form II's or equivalent for SW-846 methods included in the data package?	___	
12. Are surrogate recoveries included for all samples, standard mixtures, PEMs, blanks, and matrix spikes?	___	
13. Have the contract-specified surrogate standard concentrations been used?	___	
Matrix Spike / Matrix Spike Duplicates		
14. Are Form III or equivalent for SW-846 methods included in the data package?	___	
15. Have frequency requirements for the MS/MSD been satisfied?	___	
QC Check Standard (SW-846 only)		
16. Is a report for the QC Check Standard for SW-846 methods included in the data package?	___	
17 Was the QC Check standard both prepared and analyzed?	___	
Pesticide Cleanup Check		
18. Are Form IX or equivalent for SW-846 methods included in the data package?	___	
CRQLs and SQLs		
19. Are Form I's or equivalent for SW-846 methods included in the data package?	___	
20. Are all SQLs ≤ the RDL?	___	
21. Are all compounds detected or not detected below the SQL qualified "J" by the laboratory?	___	

Action: Indicate non-correctable contract compliance deviations below. If SQL>RDL, reanalysis may be warranted. Specify compounds that will require "J" qualification as stated in #3 above.

Attachment C
DATA VALIDATION REPORT
Page 1 of 12

Pesticide and PCB Data Validation Report

Sample Delivery Group #:

Matrix:

Laboratory:

Laboratory SOW#:

This checklist is to be used for validation of Pesticide/PCB data packages, consistent with the ES-B-0811. This checklist should be used only if the Pesticide/PCB Verification Report has been completed.

Instructions: If criteria are not met in this checklist, follow qualification provided in the "Q" column accompanied with the corresponding reason code in the "C" column. If necessary, additional codes may be placed. Sections titled "Raw Data Confirmation" need only be performed if necessary, as determined through the DQO process.

NOTE: All laboratory contractual criteria apply to both chromatographic columns; however, results should be qualified based on quality considerations of the quantification column. If quality deficiencies occur on the confirmation column, do not qualify reported results.

Validated by:_____ Date:

Attachment C
DATA VALIDATION REPORT
Page 2 of 12

1.0 Custody of Samples

Validation Step
Action: For any samples not traceable upon inspection of verification report and chains of custody, indicate the identities below and where the break in traceability occurred. Qualify samples not traceable "R (E01)." Indicate which samples were affected below.
Qualified Sample

Attachment C
DATA VALIDATION REPORT
Page 3 of 12

2.0 Holding Times and Sample Preservation

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Were all samples analyzed within the appropriate holding time?			J	UJ/R	H01, H02, H03, H04
2. Were all samples preserved properly?			J	UJ	H05

Action: Indicate affected samples below.

Sample	Holding Time Exceedence (Days)	Improper Preservation (check)
_____	_____	
_____	_____	
_____	_____	
_____	_____	
_____	_____	
_____	_____	
_____	_____	
_____	_____	
_____	_____	
_____	_____	

Comments:

Attachment C
DATA VALIDATION REPORT
Page 4 of 12

3.0 GC/ECD Instrument Performance Check

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. The performance check has been performed at the proper frequency.			*	*	C06
2. Is Resolution Check Mixture ≥ 60% on both columns?			J	R	C09
3. Is PEM resolution for all peaks is ≥ 90%?			J	R	C09
4. Do all PEM compounds fall within retention time windows?			See specific guidance in ES-B-0811.		C14
5. Is RPD between true and calculated amounts for PEM within ± 25%?			J	UJ	C15
6. Is % breakdown of 4,4'-DDT and Endrin<20%?			See specific guidance in ES-B-0811.		C16, C17
7. Is combined % breakdown of 4,4-DDT and Endrin < 30%?					
Raw Data Confirmation					
3 Chromatograms do not show peaks > CRQL and near or within retention time window.			See specific guidance in ES-B-0811.		

Action: Indicate affected samples only if data are rejected.

Sample Reason for Rejection

_____

_____

_____

_____

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

Attachment C
DATA VALIDATION REPORT
Page 5 of 12

4.0 Initial Calibration

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Was the correct calibration sequence followed?			*	*	C03
2 Were the appropriate number of standards used?			*	*	C24
3 Were the appropriate standard concentrations used?			*	*	C18
4 Was resolution of midpoint INDA and INDB ≥90%?			J	R**	C09
5. Is %RSD criteria satisfied for single-component compounds?			J	UJ	C13
Raw Data Confirmation					
6. Are recorder deflections from at least one chromatogram ≥ 50%.			See specific guidance in ES-B-0811.		C31

Action: Indicate affected samples only if data are rejected.

Sample Reason for Rejection

_____
_____
_____
_____
_____
_____
_____
_____

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.
** Qualify as appropriate

Attachment C
DATA VALIDATION REPORT
Page 6 of 12

5.0 Continuing Calibration

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Was continuing calibration performed at the appropriate frequency?			*	*	C06
2. Was resolution of midpoint INDA and INDB ≥90%?			J	R**	C09
3. Is RPD between true and calculated amount for single component compounds in INDA and INDB ≤ 25%?			J	UJ	C15
4. For SW-8081A, are response factors within ±15% of the initial calibration?			J	UJ	C13

Action: Indicate affected samples only if data are rejected.

Sample Reason for Rejection

_____
_____
_____
_____
_____
_____
_____
_____

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.
** Qualify "R" as appropriate considering other QC in the data package.

Attachment C
DATA VALIDATION REPORT
Page 7 of 12

6.0 Method Blank

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Have method blanks been analyzed at the appropriate frequency?			*	*	B05
2. Sample results are > CRQL and > 5* X Blank result					
sample result is > CRQL and < 5* X Blank result			U	n/a	B01
sample result is < CRQL and < 5* X Blank result			U (report SQL)	n/a	B01
gross contamination			R	**	B03
3. Instrument blanks have been analyzed after samples showing high concentrations.			**	n/a	B08
Raw Data Confirmation					
4. Confirm from raw data that compounds reported in the method blank are detected above the CRQL.					
Action: Indicate affected samples only if data are rejected.					

Sample Reason for Qualification

_____

_____

_____

_____

_____

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

** Qualify as appropriate

Attachment C
DATA VALIDATION REPORT
Page 8 of 12

7.0. Surrogate Standards

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Have the proper surrogate standards been used?			*	*	S04
2. Have the proper surrogate concentrations been used?			*	*	S05
3. The following checks are applicable to % recovery:					
Surrogate standards have been evaluated					
%R is 10-30%			J	UJ	S02
%R is 0-10%			J	R	S03
%R is 0			R	R	S03
%R is >150%			J	n/a	S01
4. Are surrogate standards within the appropriate retention time windows?			J	J/R	S08
Action: Indicate affected samples only if data were rejected.					

Sample Reason for Qualification

_____
_____
_____
_____
_____

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

Attachment C
DATA VALIDATION REPORT
Page 9 of 12

8.0 Matrix Spike/Matrix Spike Duplicate

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Was the MS/MSD analyzed at the appropriate frequency?			*	*	M04
2. Are all MS/MSD compounds within control criteria?			**	**	M01, M02
3. Are all MS/MSD RPD within control criteria?			**	**	M03

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	
_____	
_____	
_____	
_____	
_____	
_____	
_____	
_____	
_____	_____

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

** Qualify only after evaluating other QC data in the SDG.

Attachment C
DATA VALIDATION REPORT
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9.0 QC Check Standard (SW846 Methods)

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Was the QC Check Standard analyzed at the appropriate frequency?			*	*	K04
2. Was the QC Check Standard prepared and analyzed?			*	*	K01
3. Were the %R of the reported compounds within acceptance criteria?			J	UJ/R	K02, K03

Action: Indicate affected samples only if data are rejected.

Sample	Reason for Qualification
_____	
_____	
_____	
_____	
_____	
_____	
_____	
_____	

Comments:

* Qualify only if the deviation indicates an adverse effect on data quality.

Attachment C
DATA VALIDATION REPORT
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10.0 Pesticide Cleanup Check

Validation Step	Y	N	Q		C
			Detects	Nondetects	
1. Have frequency requirements been satisfied for Florosil and GPC cleanup?					V05
2. The following checks are applicable to % recovery:					
Pesticide cleanup has been evaluated					
% recovery is < 10%			**	R	V01
% recovery is > 120%			**	n/a	V02
Raw Data Confirmation					
3. For noncompliant cleanup analyses, raw data have been inspected for presence of high molecular weight compounds.			See specific guidance in ES-B-0811.		
Action: Indicate affected samples only if data are rejected.					

Sample Reason for Qualification

_____

_____

_____

_____

_____

Comments:

** Qualify as appropriate

Attachment C
DATA VALIDATION REPORT
Page 12 of 12

11.0 Sample Data Evaluation

Validation with Raw Data Confirmation
1. For one compound in each sample, determine whether CRQLs have been modified to SQLs.
2. Sample-specific evaluation - these evaluations are performed one sample in each batch (excluding QC).
evaluate adequacy of manual integrations of peak areas.
evaluate the presence of carryover
evaluate the validity of dilutions and reanalyses
evaluate interferences which may preclude accurate quantitation of compounds.
evaluate the eluation of compounds within the appropriate retention windows
evaluate occurrences of peak interference
evaluate pattern-matching of multi-component compounds
3. Manually recalculate 2 detected and 2 nondetected results from a selected sample in the SDG.
4. Did recalculation confirm reported results? If not, increase the frequency of recalculations until adequate confidence is gained in the reported results.
5. Confirm that samples were analyzed on GC/MS when concentration of any compound exceeds 10 ng/µl
6. Confirm that single-peak results agree within 20% RPD between column analyses.
Action: Indicate instances of manual calculations not confirming reported results. Where samples have been reanalyzed, and both reanalyses are included in the data package, indicate on the laboratory reporting forms which results are the most reliable.
Description of Sample-specific Evaluation Problems

Attachment D
QUALIFIERS AND REASON CODES
Page 1 of 4

Data Validation Qualifiers and Reason Codes

Pesticide/PCB Analytical Data

Reason codes must be used with all qualifiers placed on analytical data.

U Analyte compound or nuclide considered not detected above the reported detection limit

J Analyte compound or nuclide identified; the associated numerical value is approximated

UJ Analyte, compound or nuclide not detected above the reported detection limit, and the reported detection limit is approximated due to quality deficiency.

R Result is not usable for its intended purpose

= "Equals" sign, indicates that no qualifier is necessary

DATA VALIDATION QUALIFICATION CODES

Pesticide/PCB Analytical Data

Blanks

B01 Concentration of contaminant in the blank at a level ≥ the qualification level.
B02 Blank was not the same matrix as the analytical samples.
B03 Gross contamination exists.
B05 Blanks were not analyzed at appropriate frequency.
B07 Blank data not reported.
B08 Instrument blank not analyzed after high level sample
B09 Other (describe in comments)

Calibration

C03 Initial calibration sequence was not followed as appropriate.
C06 Calibration or performance check was not performed at the appropriate frequency.
C07 Calibration data not reported
C08 Calibration not performed.
C09 Chemical resolution criteria were not satisfied.
C10 Calibration standard matrix not the same as sample matrix
C11 Compounds quantitated against inappropriate standard or standard concentration level
C13 Calibration factor RSD criteria were not satisfied
C14 Retention time of compound outside window.
C15 PEM or INDA or INDB RPD criteria were not satisfied
C16 Breakdown of endrin or DDT was >20%.
C17 Combined breakdown of endrin or DDT was >30%.

Attachment D
QUALIFIERS AND REASON CODES
Page 2 of 4

C18 Inappropriate standard concentrations
C24 Standard curve was established with fewer than the appropriate number of standards
C31 Chromatogram does not show adequate gain setting
C32 Other (describe in comments)

Laboratory Duplicate

D02 Laboratory duplicate was not analyzed at the appropriate frequency
D03 Laboratory duplicate exceeds RPD criteria.
D04 Laboratory duplicate data not reported
D05 Other (describe in comments)

Evidentiary Concerns

E01 Custody of Sample in Question.
E02 Standard not Traceable.
E03 Other (describe in comments)

General

G01 Professional judgement was used to qualify the data.
G02 Other (describe in comments)

Holding Times/Preservation

H01 Extraction holding times were exceeded.
H02 Extraction holding times were grossly exceeded.
H03 Analysis holding times were exceeded.
H04 Analysis holding times were grossly exceeded.
H05 Samples were not preserved properly.
H06 Sample preservation can not be confirmed
H07 Sample temperature exceeded criteria prior to preparation
H08 Other (describe in comments)

Internal Standards

I01 Area count was above upper control limits.
I02 Area count was below lower control limits.
I03 Extremely low area counts or performance was exhibited by a major drop off
I04 Internal standard retention time varied by more than 30 seconds
I05 Inappropriate internal standard used
I06 Inappropriate internal standard concentration(s) used
I07 Internal standard data not reported.
I08 Other (describe in comments)

Attachment D
QUALIFIERS AND REASON CODES
Page 3 of 4

QC Check Standard

K01 SVOA or PEST/PCB QC Check Standard not analytically prepared, but only analyzed
K02 Recovery of QC Check Standard was above upper control limits
K03 Recovery of QC Check Standard was below lower control limits
K04 QC Check Standard data not reported
K05 Other (describe in comments)

Matrix Spike and MS/MSD

M01 MS and/or MSD recovery above upper control limit.
M02 MS and/or MSD recovery below lower control limit.
M03 MS/MSD pair exceeds the RPD limit.
M04 MS and/or MS/MSD not analyzed at the appropriate frequency
M05 MS and/or MS/MSD data not reported
M06 Other (describe in comments)

Instrument Performance

P02 Extraneous peaks were observed.
P03 Loss of resolution was observed.
P04 Peak-tailing or peak splitting that may result in inaccurate quantitation were observed
P07 Other (describe in comments)

Quantitation

Q01 Peak Misidentified
Q02 Target analyte affected by interfering peak.
Q03 Qualitative criteria were not satisfied.
Q04 Cross contamination occurred.
Q05 Second column confirmation not run.
Q06 GC/MS confirmation needed but not done.
Q08 The %D between the two pesticide/PCB column checks was >25%
Q12 No raw data were provided to confirm quantitation.
Q14 Inappropriate aliquot sizes were used.
Q18 Compounds were not adequately resolved.
Q24 Raw data anomaly
Q25 Other (describe in comments)

Surrogates

S01 Surrogate recovery was above the upper control limit.
S02 Surrogate recovery was below the lower control limit.
S03 Surrogate recovery was <10%.
S04 Inappropriate surrogate standard used
S05 Inappropriate surrogate standard concentration(s) used
S06 Surrogate data not reported.

Attachment D
QUALIFIERS AND REASON CODES
Page 3 of 4

S07 Surrogate outside retention window
S08 Other (describe in comments)

Pesticide Cleanup

V01 10% recovery or less was obtained during either check.
V02 Recoveries during either check were >120%.
V03 GPC Cleanup Not Performed on Soil or Affected Water Sample.
V04 Cleanup data not reported
V05 Cleanup check not performed at the appropriate frequency
V06 Other (describe in comments)

Attachment E
PESTICIDE/PCB VALIDATION IMPLEMENTATION FORM
Page 1 of 1

Pesticide/PCB Data Validation Implementation Form

Purpose: This worksheet is the mechanism to be used to identify and document the implementation of procedure ES-B-0811.

Instructions: One worksheet must be completed for each fraction and analytical method (e.g. Pesticides, method 8081A). The completed worksheet shall be forwarded to the ER Data Quality Program for approval.

If the requirements of ES-B-0811 are adopted as-is, without the necessity of approved alternatives, enter "as-is" for review item "A" in the implementation column and complete the project sign-off section at the bottom of the form. No other entries are needed.

If project-specific implementations are necessary,

1) identify review items that will be directly implemented and enter "as-is" in the respective rows of the Implementation column.
2) identify review items that will not be considered by verifiers or validators during the implementation of this procedure, and enter "n/a" in the respective rows of the Implementation column and reference attached materials documenting the reason for the exclusion of these review items in the Comment column.
3) Identify review items that will be implemented with project-specified alternative actions and enter "Alternative" in the respective rows of the Implementation column. In the Comment column, reference attached materials that document the need for the alternative actions and specify the actions to be implemented upon approval. This alternative description should cover the following: Deliverables, Frequency, Performance Criteria, Verification step(s), Validation step(s).
4) Complete the project sign-off section at the bottom of the worksheet and forward to the ER Data Quality Program for approval.

Fraction and Method:

	Implement	Comment
1. All requirements of ES-B-0811 will be implemented	____	
2. Technical Holding Time	____	
3. GC/ECD Instrument Performance Check	____	
4. Initial calibration	____	
5. Continuing Calibration	____	
6. Blanks	____	
7. Surrogate Standards	____	
8. Matrix Spikes / Matrix Spike Duplicates	____	
9. QC Check Standard	____	
10. Pesticide Cleanup Check	____	
11. CRQLs and SQLs	____	
12. Manual Calculation of Results	____	

The _____ project will approach pesticide/PCB validation consistent with ES-B-0811, and/or with specific alternative(s) described on the attached pages.

_____

Signature Date

Attachment F
QUALIFICATION TABLES FOR MULTIPLE QUALITY DEFICIENCIES
Page 1 of 1

Guidance for Data Qualification

Due to Multiple Quality Deficiencies

This appendix provides guidance in the qualification of data due to instances of multiple quality deficiencies. Quality deficiencies can be categorized based on potential effect on sample data. The effect of quality deficiencies may be applicable to only a single sample or to all samples within the reporting batch. A validation qualifier and associated reason code(s) should not be placed on sample data until all quality deficiencies have been identified within the reporting batch.

The following is a listing of data quality indicators and the probable effects on sample data.

Data Quality Indicator	Effect on Sample Data
GC performance check	identification and quantitation
Initial calibration RSD	quantitation
Continuing calibration	quantitation
Method blank	positive bias
Surrogate standards	positive or negative bias
Matrix spike / matrix spike duplicate	positive or negative bias and precision
QC check standard	positive or negative bias
Florosil cleanup	quantitation
GPC cleanup	quantitation

In the instance of multiple quality deficiencies the validation qualifier and reason code(s) should be placed consistent with the acceptable level of uncertainty associated with the intended use of the data. The validation SOW should provide a summary of the intended use(s) of the data. (e.g., risk assessment, fate and transport modeling, waste management) to facilitate appropriate placement of validation qualifiers.

Attachment G
CALCULATIONS AND EQUATIONS
Page 1 of 2

Rounding rules

1. In a series of calculations, carry the extra digits through to the final result, then round off.

2. If the digit to be removed is less than 5, the preceding digit stays the same.

   If the digit to be removed is equal to or greater than 5, the preceding digit is increased by 1.

G.1 MS Percent Recovery

$$\%R_{MS} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spiked sample result
SR = Sample result
SA = Spike Added

G.2 Relative Percent Difference

$$RPD = \frac{|R1 - R2|}{\overline{X}_{(R1,R2)}} \times 100$$

R1 = Result 1
R2 = Result 2

G.3 Sample Quantitation Limit

$$SQL = CRQL_{SOW} \times DF \times S \times \frac{BW}{SW} \times \frac{BA}{SA} \times \frac{1}{\%S}$$

$CRQL_{sow}$ = contract required quantitation limit from the CLP SOW 3/90
DF = dilution factor
%S = percent solids (100 - % moisture)/100
S = splitting factor (for sample volumes between column analyses)
BW = Method Blank weight
SW = Sample Weight
BA = Method Blank Aliquot
SA = Sample Aliquot

Attachment G
CALCULATIONS AND EQUATIONS
Page 2 of 2

G.4 Results for Waters

$$\mu g/L = \frac{A_X \times V_T \times D_F}{C_F \times V_O \times V_I}$$

A_x = Area of measured compound peak
CF = Calibration Factor for midpoint concentration (area per ng)
V_o = Volume of water extracted in mL
V_i = Volume of extract injected in µL (use 1/2 volume if single injection is made onto two columns
V_t = Volume of the concentrated extracted (must be 10000 µL)
D_f = Dilution Factor

G.5 Results for Soils/Sediments (Dry weight basis):

$$\%R = \frac{A_X \times V_t \times D_f \times 2.0}{CF \times V_i \times W_s \times \%S}$$

A_x = Area of measured compound peak
CF = Calibration Factor for midpoint concentration (area per ng)
V_i = Volume of extract injected in µL (use 1/2 volume if single injection is made onto two columns
V_t = Volume of the concentrated extracted (must be 5000 µL)
D_f = Dilution Factor
%S=(100-% moisture)/100
W_s = Weight of sample extracted in grams

G.6 Surrogate Percent Recovery

$$\%R = \frac{S_{ng/g}}{\dfrac{(C_s V_s \times 1000 ng/\mu g)}{Wg \times \left(100 \times \dfrac{M}{100}\right)}} \times 100$$

$S_{ng/g}$ = Surrogate concentration
V_s = Volume of surrogate solution spiked into analytical sample (0.1 ml for waters; 0.2 ml for soils
C_s = Concentration of surrogate solution (2 ug/ml)
W_g = Sample Weight in grams
M = % Moisture (for soils only)

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED:

Environmental Restoration, Waste Management
Procedure Type
(Company, Functional, Project, Subcontractor)

GEO-TEC-007, Data Management Coordination
(Procedure Number and Title)

11/03/2003
(Procedure Date)

2
(Rev. Number)

Reason for Changes: Transitioning of BJC Procedures

SECTION	DESCRIPTION OF CHANGE
General	Replace Bechtel Jacobs Company LLC (BJC) with Paducah Remediation Services LLC (PRS)

Initiator/Originator: Darryl Potter / [signature] — 4/7/06
(Print Name / Sign Name) — Date

REVIEWED BY: [signature] — 4/7/06
Discipline/Task Manager — Date

APPROVED BY: Diane McDaniel — 4/7/06
Project Manager — Date

BJC to PRS Position/Title/Organization Matrix
for GEO-TEC-007 Data Management Coordination

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
Project Manager	→	Project Manager
Project Data Coordinator	→	Data Entry Specialist
QA Coordinator	→	Quality Assurance
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PURPOSE

1.1 To describes the procedures and interfaces with Bechtel Jacobs Company LLC (BJC) for organizing and maintaining the integrity of data generated at the Paducah site.

1.2 Databases are utilized in an integrated data management system so that data can be sorted by contaminant type, contaminant concentration, study area, date, sample depth, etc.

NOTE: A project data management plan may be prepared. The data management plan describes specific procedures for field and analytical data storage and retrieval, data summarizing and reporting, data life cycle, data security, and back up.

SCOPE

2.1 This procedure applies to all personnel who perform work activities for GEO Consultants, LLC (GEO) and its subcontractors under contract to BJC at the U.S. Department of Energy (DOE) owned Paducah Site. No changes or deviations to this procedure are allowed unless approved in advance by the BJC Subcontract Technical Representative (STR) before activity implementation.

TRAINING

3.1 Required Reading

OTHER DOCUMENTS NEEDED

4.1 GEO-TEC-003, "*Records Management*"

4.2 GEO-TEC-004, "*Quality Assured Data*"

4.3 GEO-TEC-008, "*Sample Tracking and Handling Guidance*"

WHAT TO DO

NOTE: Definitions and acronyms are provided in Attachment A.

5.1 Data Management

Project Manager

5.1.1 Assign Project Data Coordinator for the project.

Project Data Coordinator

5.1.2 Assist Sample Manager with inputting required sample information to pre-load the Paducah Project Environmental Measurements System (Paducah PEMS).

5.1.3 Enter data into Paducah PEMS as required by GEO-TEC-008, "*Sample Tracking and Handling Guidance*," and other non-sample information required by Paducah PEMS (e.g., lithologic descriptions, well construction, etc.).

5.1.4 Verify data entry by the single-data entry with the 100% visual verification method. Data entry should be printed from Paducah PEMS and checked against the original by someone other than the enterer if at all possible.

5.1.5 Document the verification electronically in Paducah PEMS or on the hardcopy printouts and maintain the documentation as quality control (QC) records.

5.1.6 **IF** Geographical Information Systems (GIS) coverage is created for the project, **THEN** ensure the format is compatible with the Paducah GIS structure.

NOTE: Arc/Info is preferred.

Project Data Coordinator

5.1.7 **WHEN** laboratory electronic data deliverables (EDDs) are received from BJC, **THEN** load them into Paducah PEMS.

5.1.8 Verify the first 4 EDDs loaded to Paducah PEMS and then 10% of subsequent EDDs loaded by comparing the laboratory printouts or data packages to printouts of data loaded to Paducah PEMS.

NOTE: Laboratory printouts/data packages are available from the BJC Sample Manager.

5.1.9 Record verification in the electronic Paducah PEMS Loading Log under the Errors Noted field.

5.1.10 **IF** discrepancies are found,
THEN notify the BJC Data Manager and/or BJC sample manager immediately.

5.1.11 **WHEN** data is loaded to Paducah PEMS,
THEN verify the data according to GEO-TEC-004, "*Quality Assured Data*," and applicable project-specific operator aids.

5.1.12 Ensure the following is documented in the electronic Paducah PEMS loading logs and/or in Paducah PEMS loading notes sent via e-mail and included in the data assessment package:

- Project Identification
- Statement(s) of Work Number(s)
- Date Loaded
- Number Records Loaded
- Description
- Electronic Deliverable Origin
- Errors Noted (verification performed)

QA Coordinator

5.1.13 Submit data packages and laboratory resubmissions to the data validator.

Project Data Coordinator

5.1.14 Upon receipt of validation codes, load/enter codes into Paducah PEMS.

5.1.15 Return data package to the BJC Sample Manager.

5.1.16 Verify entry according to step 5.1.4.

5.1.17 Place a copy of the validation report in the data assessment package.

Project Team

5.1.18 Assess project data in accordance with GEO-TEC-004, "*Quality Assured Data*."

Project Data Coordinator

5.1.19 **WHEN** data assessment is complete,

Attachment A
Definitions and Acronyms
Page 1 of 2

1.0 Definitions

1.1 **Bechtel Jacobs Company Data Manager** - The BJC Data Manager will interface with the GEO Data Manager to oversee PEMS usage and to ensure that data deliverables meet BJC's standards. The BJC Data Manager will enter information related to the fixed-base laboratory data packages and the tracking associated with the samples, once the samples have been shipped from the laboratory and the BJC Sample Manager has verified receipt of samples. The BJC Data Manager also is responsible for transferring project data to the Paducah OREIS database from the ready-to-load (RTL) files supplied by GEO.

1.2 **Bechtel Jacobs Company Sample Manager** - The BJC Sample Manager will develop the statement of work (SOW) to be performed by an analytical laboratory in the form of a project-specific laboratory SOW. Analytical methods, detection limits, minimum detectable activities, laboratory QC requirements, and deliverable requirements will be specified in the SOW. The BJC Sample Manager will provide GEO with a matrix template. The GEO PEMS Manager will enter the sampling requirements and submit the completed and approved sampling matrix to the BJC Sample Manager to assist in developing the laboratory SOW. The BJC Sample Manager will receive EDDs, perform contractual screenings, and distribute data packages. The BJC Sample Manager will interact with the GEO Data Manager to ensure that hard copy and electronic-deliverable formats are properly specified and will interface with the contract laboratory to ensure that the requirements are understood and met.

1.3 **Data** - A formalized representation of facts or concepts suitable for communication, interpretation, or processing by people or by automatic means; the raw material of information; individual pieces of quantitative information ("data" is the plural of "datum" but the singular form of this word is rarely used in common speech).

1.4 **Data Assessment Package** - Includes complete forms, PEMS loading notes, data verification results, and data validation reports.

1.5 **Data Integrity** - The accuracy, consistency, and completeness of the data that are maintained by a computer system.

1.6 **Data Management** - **(1)** A general term that collectively describes those functions of a system that provide access to hardware, enforce data storage conventions, and regulate the use of input/output devices; **(2)** A major function of operating systems that involves organizing, cataloging, locating, retrieving, storing, and maintaining data.

1.7 **Environmental Data** - Any measurements or information that describes environmental processes or conditions, or the performance of environmental technologies. (ANSI/ASQC E4 - 1994)

1.8 **Geographic Information System** - Location information designed to display data spatially.

1.9 **Integrated Data Management System** - The collection of software and hardware that organizes and provides access to data. All aspects are integrated and complement the other components.

1.10 **Paducah Oak Ridge Environmental Information System** - The Paducah database system used for long-term storage of data. Paducah OREIS is the primary database system used for official data reporting.

1.11 **Paducah Project Environmental Measurements System -** The Paducah database system used for field preparation and tracking of data collection activities. This system is provided to project personnel and pre-populated with project information. The system generates chains-of-custody, bottle labels, and other field forms; stores analysis type and preservative information; tracks' sampling progress; and stores location information. Used to load analytical data verification and assessment of analytical data.

1.12 **Project Data Coordinator** -The Project Data Coordinator assists the BJC Sample Manager in pre-populating Paducah PEMS. The Project Data Coordinator is responsible for loading electronic data deliverables provided by BJC, electronic verification of data, and tracking the data assessment process. The Project Data Coordinator is the primary contact for all field activities relating to data and works closely with the BJC Sample Manager and the BJC Data Manager.

1.13 **Project Manager -** The Project Manager is responsible for coordinating sample collection, sample analysis, data assessment, and decision-making.

1.14 **Project Team -** The personnel involved in the data collection process of planning, review, and decision-making. The team includes, but is not limited to, the Project Manager, QA Coordinator, and the Project Data Coordinator.

1.15 **QA Coordinator** -The Project Team member involved in the planning and review of data to ensure that data quality requirements are met. The QA Coordinator works closely with the BJC Data Manager and the BJC Sample Manager.

2.0 Acronyms

2.1 **DOE** - Department of Energy

2.2 **EDD** - Electronic Data Deliverable

2.3 **GIS** - Geographic Information System

2.4 **OREIS -** Oak Ridge Environmental Information System

2.5 **Paducah PEMS** - Paducah Project Environmental Measurements System

2.6 **STR** - Subcontract Technical Representative

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Work Control, Engineering, and Facilities	**PRS-DOC-1009**	**REV. NO. 0**
SUBJECT MATTER AREA: Documents	**PREPARER: Sharon Hankins**	**Page 1 of 10**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:**	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**	**Tim Fralix (Signature on File in DCC)** **5/10/07**	
TITLE: **RECORDS MANAGEMENT, ADMINISTRATIVE RECORD, AND DOCUMENT CONTROL**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 6/10/07**	
USQD/UCD No: UCD-PH-SITE-0099/USQD-PH-SITE-0065	**REQUIRED REVIEW DATE: 06/10/10**	
Mandatory Subcontractor Pro Forma Procedure? ☒	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. This document replaces PRS blue-sheeted procedures BJC-OS-1003, BJC-LE-1002, and BJC-OS-1001.	All

CAUTION

This procedure describes specific safety basis requirements for safety significant items used by the Paducah Project. Any proposed changes must be processed in accordance with the procedure change control process defined in PRS-DOC–1107/R1 and all changes shall be reviewed by the USQD process and approved by PRS Nuclear/Facility Safety. Specific safety basis requirements are noted in this document in the following format: Specific TSR requirements are noted in the document as <TSR, Section Number, and Title (if applicable)>.

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1.0 PURPOSE

This procedure establishes consistent administrative controls and assigns responsibility and defines protocols for the identification, control, and management of documents and records. This procedure also establishes the requirements for transmitting non-Federal Facility Agreement (FFA) Documents and Administrative Records to the Paducah Remediation Services, LLC (PRS) Document Control Center (DCC).

2.0 SCOPE

This procedure applies to all PRS personnel and subcontractor employees who create, process, or use records for the U. S. Department of Energy (DOE). This applies to all types of record media.

PRS document procedures govern the scope of the PRS Document Program. PRS issues controlled project-specific procedures and documents for the performance of work. The DCC is the control center for documents being issued as a PRS document.

3.0 TRAINING

This procedure is required reading for all PRS personnel and PRS subcontractor personnel who request documents or submit documents to the DCC.

4.0 PROCEDURE

4.1 Creating and Identifying Records

4.1.1 Records are books, papers, photographs, machine readable materials, maps, or other documentary materials, regardless of physical form or characteristics, which have documentary or evidential value. Such materials, created or received in connection with the transaction of official business, are preserved because of their informational value as evidence of the organization, functions, policies, decisions, procedures, operations, or other activities <Facility Specific TSR, 5.9>.

Preparer

4.1.2 Create a document/record that adequately and properly documents the work performed, ensuring it is:

- Accurate, legible, reproducible, and complete
- Developed according to appropriate guidelines
- Validated by appropriate personnel

4.1.3 For correspondence numbers only, go the PRS Correspondence Log housed at S:\Everyone\PRS Correspondence Log.

4.1.4 For document numbers, contact the DCC for appropriate number.

4.1.5 **IF** it is a non-FFA Document/Record, **THEN** continue to Section 4.2.

4.1.6 **IF** it is an Administrative Record, **THEN** go to Section 4.3.

4.1.7 **IF** it is a Controlled Copy, **THEN** go to Section 4.4.

4.2 Non-FFA Documents/Records

Preparer

4.2.1 Make distribution of your document/record ensuring that the DCC/Document Management Center (DMC)-RC (Record Copy) is on distribution for a hard copy and electronic copy of all documents/records.

4.2.2 If a record/document is to be forwarded to the Swift & Staley Team (SST) DMC, complete the SST Material Transfer Form (MTF), SSTF-011, (Attachment B), Classification and Clearance Review Form (SSTF-106), or Records Transmittal List (SSTF-12) for boxes, and submit to the DCC.

DCC

4.2.3 Receive the document/record, ensure all information is complete, log into the system, and date stamp document/record.

4.2.4 Submit to the SST DMC for processing and proper storage.

4.2.5 After document/record has been processed by the SST DMC and a copy of the MTF has been returned to DCC staff, log accession number into correspondence database.

4.2.6 **IF** the document/record is PRS proprietary, **THEN** log into the DCC system, prepare folder, and file in the PRS DCC. **DO NOT** forward a copy to the SST DMC.

4.3 Administrative Records (AR)

4.3.1 Contents of the Administrative Record include a variety of written material, such as pieces of correspondence, data reports, assessments, plans, newspaper articles, notices, and fact sheets. Also included, but not limited to, are archive search reports, site photographs and maps, site descriptions and chronologies, reference documents, sampling and analysis data and plans, work plans, site safety and health plans, applicable or relevant and appropriate requirements, engineering evaluation/cost analyses, remedial investigation/feasibility studies, health and endangerment assessments, proposed plans for remedial action, records of decision, community relations plans, public meeting minutes/transcripts, environmental baseline studies, and findings of suitability to transfer/lease documents.

AR Coordinator

4.3.2 Approve initiation of an AR File for each Comprehensive Environmental Response, Compensation, and Liability Act of

1980 (CERCLA) response action at commencement of a decision-making process.

4.3.3 **COORDINATE** with Public Affairs the creation of a "Notice of Availability" for the AR File upon approval of the initial primary document for a particular response action or documentation of approval to proceed. **THEN** submit two copies of the document, cover letter, Classification and Review Form, and MTF to the DCC.

Public Affairs **4.3.4** Publish the "Notice of Availability" for the AR File in local newspaper(s) of record.

NOTE: Ensure that the DCC/DMC is the record copy holder for new or updated fact sheets, information briefs, and public notices.

Preparer **4.3.5** Ensure that the DCC/DMC-RC is included on distribution as the record copy holder to receive two copies of all Primary and Secondary Documents.

DCC **4.3.6** Receive documents, date stamp, and log into the DCC system.

4.3.7 Ensure all enclosures/attachments are with the cover letter and that appropriate classification and clearance reviews have been completed.

4.3.8 Forward the AR documents to the SST DMC for processing and entry into the on-site AR and the off-site AR Environmental Information Center.

AR Coordinator **4.3.9** Attend Administrative Record Content Completeness Meetings which are held prior to the public comment period to ensure completeness of the AR File up to that point.

4.3.10 Request an AR File Index from the SST DMC and transmit to the appropriate project manager for review and submittal to DOE during the D0 decision review.

Preparer **4.3.11** Transmit the Proposed AR File Index to DOE with the D0 decision document.

4.4 Controlled Copies

Owner **4.4.1** Contact the DCC with your request to make a document(s) a controlled copy.

DCC **4.4.2** Assign a PRS Controlled Copy number to the document and create a folder on the PRS SharePoint.

4.4.3 Upload document to the appropriate PRS SharePoint folder.

Preparer

4.4.4 Print controlled copy and cover page and follow instructions contained on the cover page.

DCC

4.4.5 Ensure that revisions/changes to controlled documents receive the same level of review and approval as the original.

4.4.6 Prepare a Historical File and maintain a list of all copies issued on controlled distribution.

4.5 Freedom of Information Act (FOIA)

4.5.1 If the DCC receives a FOIA request, inform the requester to contact the DOE FOIA officer. All FOIA requests are to be submitted to the DOE FOIA officer.

4.5.2 If the DOE FOIA officer approves the request, PRS will provide response appropriate to the request.

4.6 Requesting Records from the SST DMC

Requester

4.6.1 Send your record request to the DCC. Include a date by which you need the record.

- Give the title of the record requested
- **IF** title is not known, **THEN** list as many keywords that may help to identify the document
- Date of the record, if known
- Any other pertinent information that may benefit the DMC in locating the record

DCC

4.6.2 Complete the Records Request form (Attachment C) with all the pertinent information and send to the DMC for completion.

4.6.3 Request that the record be sent directly to the requester.

4.7 Storage of Documents

NOTE: Storage of records are to be in an approved storage vault such as the SST DMC, or

- One-hour, fire-rated cabinet, plus adequate smoke detection, or fire suppression system with reasonable safeguards against theft, water damage, and insect or rodent infiltration; or
- Maintain duplicate records in identified duplicate storage areas in separate locations. These locations should be sufficiently remote from each other to eliminate the chance of exposure to a single hazard; or
- Duplicate information in another record media and store in a separate location.

4.7.1 The DOE record schedule for environmental records provides for the retention and disposition of records in accordance with the laws and regulations of the Department of Energy New Environmental Records Schedule (15000) <Site-wide TSR, 5.8 and Facility Specific TSR 5.9>.

DCC **4.7.2** Establish access control to prevent unauthorized use, disclosure, theft, or destruction of proprietary records or field operating records (FORs).

4.7.3 Post a list designating personnel approved for access to records located in satellite areas.

4.7.4 No records may be removed from the satellite records area without the approval of the DCC manager.

4.7.5 Copies of FORs may be made for individuals with the need-to-know.

4.8 Requesting Document Numbers

Requester **4.8.1** See procedure PRS-DOC-1004 for the types of document numbers supplied by the DCC.

4.8.2 Call the DCC for your document number.

DCC **4.8.3** Provide appropriate document number to requestor.

5.0 RECORDS - RESERVED

6.0 SOURCE DOCUMENTS

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in brackets will become the reference procedures once these procedures are approved and implemented by Paducah Remediation Services, LLC.

- PRS Blue-Sheeted Procedure BJC-OS-1003 [PRS-DOC-1009], *Administrative Record*
- PRS Blue-Sheeted Procedure BJC-OS-1001, *Records Management, Including Document Control* [PRS-DOC-1009]
- PRS Procedure PRS-DOC-1004, *Document Numbering and Issuance*
- Office of Solid Waste and Emergency Response (OSWER), Directive 9833.3A-1, U.S. Environmental Protection Agency, Washington, D.C. *Final Guidance on Administrative Records for Selecting CERCLA Response Actions*
- *Department of Energy New Environmental Records Schedule (15000)*

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 1

DEFINITIONS

ADMINISTRATIVE RECORD - The Administrative Record serves two primary purposes. First, the record contains documents which form the basis for selection of a response action. Section 113 (j)(1) of CERCLA and general principles of administrative law, limits the courts to the use of the Administrative Record to support judicial review when the adequacy of a response action is being challenged. Second, the record acts as a vehicle for public participation, thus ensuring that the government has considered the concerns of the public during the response selection process.

CONTROLLED DOCUMENT REQUESTER - The person who requests that a specific document be made into a controlled document by the DCC for use in their group.

OWNER - The owner is the senior manager in the owning organization. Owners are the final approval authority for all procedures developed by a specific project, or project support, organization.

PREPARER – The individual, assigned by the Facility Manager/Paducah Remediation Manager of Projects or designee, who is responsible to facilitate and coordinate development, comment resolution, and approval of a Performance Document.

REQUESTER – The individual who is requesting the document or record, number, etc., from the DCC.

ACRONYMS

AR – Administrative Record
AR Coordinator – The individual who approves the initiation of an AR file.
CERCLA – Comprehensive Environmental Response, Compensation, and Liability Act of 1980
DCC – Document Control Center that is controlled by PRS
DMC – Document Management Center that is controlled by Swift & Staley
DOE – United States Department of Energy
FOIA – Freedom of Information Act of 1966
MTF – Material Transfer Form
PRS – Paducah Remediation Services, LLC
RC – Record Copy
RCRA – Resource Conservation and Recovery Act of 1976
SST – Swift & Staley Team

Attachment B
MATERIAL TRANSFER FORM
Page 1 of 1
This is only an example of the form. Contact Document Control for usable form.

PADUCAH PROJECTS
Document Management Center - Material Transfer Form

Document Title:	Document Date:
Document No.	Page Count:
Author/Organization:	Facility/Operable Unit:

WASTE-RELATED RECORDS:

Waste Shipment Records:	Waste Disposition File Groups: (check one below if applicable)	Waste Operations File Groups: (check one below if applicable)
Shipment Number *(required)*:	Miscellaneous ☐	Waste Storage ☐
PDL#: _____ ***OR*** PAD#: _____	RCRA Waste ☐	Landfill Operations ☐
OSSMR#:_____ Profile#:_____	TSCA Waste ☐	PCB Activity ☐
Hazardous Waste Manifest# (if applicable): _____	Low Level Waste ☐	

CLASSIFICATION OFFICE/ DERIVATIVE CLASSIFIER (DC) TO COMPLETE:

Classification: **Classified**_____ *(level & category)* **Unclassified** _____

Reviewed for Classification: _____ *DC Name/Initials* _____ *Date Reviewed*

Key words/identifiers/buzz words: (Note: please select words from body of document).

1. _____ 3. _____ 5. _____
2. _____ 4. _____ 6. _____

Document Submitted by: _____ Date _____ Phone _____

Signature: _____

Portion Below To Be Completed By DMC Staff

Notes:

Label:	Status:	Location:	Vital Record:	Quality Record:
			Yes ☐ No ☐	Yes ☐ No ☐

FG:_____ PJ/RS: _____ Item #: _____

DMC staff initial and date:

Indexed by: _____ Date: _____ Attributed by: _____ Date: _____

Scan: Yes ☐ No ☐ QA Check (Completed after scanned) 1. ☐ 2. ☐

SSTF-011(Revised 1/06)

Attachment C
RECORDS REQUEST FORM
Page 1 of 1
This is only an example of the form. Contact Document Control for usable form.

Swift and Staley
RECORDS REQUEST FORM

Requester Name (Last, First Initial, Middle Initial)		Date of Request:	Date Required By:
Badge:	Phone:	Charge Number (if applicable):	
Company: PRS			
Purpose of Release:			

PLEASE FILL IN ANY KNOWN INFORMATION:

Document Number	Title	Author(s)	Date
1.			
2.			
3			
4.			
5.			

Special Instructions

Number of Copies Per Document:	1.	2.	3.	4.	5.

AUTHORIZED BY
(Functional Manager, Subcontract Technical Representative, etc.)

Name (Last, First Initial, Middle Initial)	Signature:	Date:
Title:		

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Completed By:	Date Completed:

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED:

Function - Procedure EM
Procedure Type
(Company, Functional, Project, Subcontractor)

PA-5003: Quality Assured Data - Paducah
(Procedure Number and Title)

8/31/2004
(Procedure Date)

Rev.0, Change A
(Rev. Number)

Reason for Changes: Transitioning of BJC Procedures to PRS INTERIM DOCUMENT

SECTION	DESCRIPTION OF CHANGE
General	When reference is made to Lockheed Environmental Management and/or Bechtel Jacobs LLC personnel or contract, change the reference to Paducah Remediation Services (LLC) personnel and contract. Note that the procedure numbers will not change until after the first revision by PRS LLC.
General	Change Bechtel Jacobs LLC Environmental Management (EM) Projects to Paducah Remediation Services (LLC) Environmental Management (EM) Projects. Change call out of Bechtel Jacobs LLC Environmental Management (EM) Projects.
Section 2.2	Delete the following sentence. "Subcontract Technical Representatives (STRs) shall ensure that subcontracts reference use of this procedure when applicable."
Attachment B - Paducah Sampling and Data Management Flowchart	The flow chart is divided by contractor and subcontractor. It is anticipated that most activities that will utilize this procedure will be mostly self performed. Some activities will be subcontracted during transition.
Table C.3 - DQO PROCESS	The DQO process presented on this table does not follow the EPA 7-step DQO process. The procedure has 5 steps and they do not correlate directly to the EPA guidance. Whenever possible, the EPA 7 step process should be utilized. It appears the EPA 7-step process will meet and exceed the procedure 5-step process.

P. 27

Initiator/Originator:	Darryl Poteet / [signature] (Print Name / Sign Name)	4/7/06 Date
REVIEWED BY:	[signature] Discipline/Task Manager	4/7/06 Date
APPROVED BY:	Diane McDaniel Project Manager	4/7/06 Date

BJC to PRS Position/Title/Organization Matrix
for PA-5003 Quality Assured Data - Paducah

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
Sample Personnel	→	Sample Technician
Paducah BJC Sample Manager	→	Environmental Monitoring/Sample Manager
Paducah BJC Data Manager	→	Data Manager/Network Specialist
Paducah BJC Quality	→	Quality Assurance
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BECHTEL JACOBS
Bechtel Jacobs Company LLC

		USQD
OWNER: Paducah Project	PA-5003	REV. NO. 0
SUBJECT AREA: Data and Sampling	EFFECTIVE DATE: 08/31/2004	Page 1 of 39
DOC TYPE: Procedure	PREPARER/POC: A. M. Bosch/A. M. Bosch	
TITLE: QUALITY ASSURED DATA - PADUCAH	APPROVED BY/DATE: G. E. VanSickle – 08/06/2004 [Signature on File] Required Review Date: 08/31/2007 (3 years)	

Administrative Procedure

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REVISION LOG		
Rev. Number	Description of Changes	Pages Affected
0	**Initial Release** – Replaces PMSA-1001, Rev. 0, Chg. A	All

PURPOSE

1.1 This procedure describes the process, including data collection and data review, to ensure *consistent* and *quality assured data* which ensures that all data released for decision making and/or external use have received adequate quality assurance reviews.

1.2 *Consistency* is provided by the use of common resources and services such as Sample Management, Data Management, a centralized data system, Quality personnel, and common definitions for data quality.

1.3 *Quality assured data* is obtained through appropriate planning, adequate sampling and laboratory quality controls, and documented data review.

SCOPE

2.1 The requirements of this procedure apply to work performed for the United States Department of Energy (DOE) at the DOE-owned Paducah Site by or for the Bechtel Jacobs Company LLC (BJC).

2.2 This procedure applies to both self-performed work by BJC employees and applicable work performed by subcontractors if required by their subcontracts. Subcontract Technical Representatives (STRs) shall ensure that subcontracts reference use of this procedure when applicable.

2.3 This procedure applies to screening and definitive data that is collected by all BJC programs/projects at Paducah. The procedure allows for flexibility in implementation for programs and projects based on data collection needs and final use of the data.

2.4 Exceptions: This procedure does **NOT** apply to:

- historical data as defined in Attachment A
- data collected by the Health and Safety Program (separate procedures define quality and management requirements)
- personnel and financial data
- access to electronic data systems (refer to Paducah BJC project procedure PA-5007 (PMIO-1003), *Access to Electronic Data Systems – Paducah*).

TRAINING

3.1 The BJC Data Manager and BJC Sample Manager shall complete and document required reading of this procedure.

OTHER DOCUMENTS NEEDED

4.1 BJC company procedure BJC-ES-1004 (ES-A-0101), *Implementing and Documenting the Data Quality Objectives (DQO) Process for Environmental Restoration (ER) Projects*

4.2 BJC company procedure BJC-OS-1001, *Records Management, Including Document Control*

4.3 Paducah BJC project procedure PA–1011, *Paducah Document Management Center – Access, Submittals, and Requests*

4.4 Paducah BJC project procedure PA-5007 (PMIO-1003), *Access to Electronic Data Systems – Paducah*

4.5 Paducah BJC project procedure PA-5100 (ERWM/ER-P2213), *Data Validation Plans for Paducah Projects*

4.6 Paducah BJC project procedure PA-5102 (ES-A-2209), *Radiochemical Data Verification and Validation - Paducah*

4.7 Paducah BJC project procedure PA-5104 (ES-B-0808), *Environmental Measurements Verification and Validation - Paducah*

4.8 Paducah BJC project procedure PA-5105 (ES-B-0810), *Volatile and Semivolatile Data Verification and Validation - Paducah*

4.9 Paducah BJC project procedure PA-5106 (ES-B-0811), *Pesticide and PCB Data Verification and Validation - Paducah*

4.10 Paducah BJC project procedure PA-5107 (ES-B-0812), *Inorganic Data Verification and Validation - Paducah*

ACTION STEPS

5.1 Initiation of Data Collection

NOTE: Attachment B provides an overall flowchart for Paducah sample and data management. Attachment C provides a flowchart on the data cycle as described in this procedure.

Requester

5.1.1 Determine need for data to support the activity or program/project.

Requester and Program/ Project Team

5.1.2 Choose the Data Quality Objective (DQO) process option for the program or project outlined in Attachment D, "Options to Implementing and Documenting the Data Quality Objectives (DQO) Process for Paducah BJC Programs/Projects."

Requester and Program/ Project Team

5.1.3 Follow steps associated with the DQO process.

5.1.4 Select Quality Assurance (QA)/Quality Control (QC) requirements using Attachment E, "Paducah BJC Data Quality Reference List" to incorporate into program/project plans.

5.1.5 Choose the data review options for the program or project using Attachment F, "Options for Data Review."

5.1.6 Prepare applicable plans, such as the Sampling and Analysis Plan (SAP), the Quality Assurance Plan (QAP), Environmental Monitoring Plans (EMP), Waste Management Plans (WMP), and Data Management Plans using Attachment G, "Estimated Timeframe for Routine Data Review Process," as a guide to establish data review schedules.

5.1.7 Complete the checklist in Attachment H, "Identification of Program/Project Data Quality Checks."

5.1.8 Notify the Paducah BJC Data Manager of electronic data quality checks that Data Management will be requested to perform.

5.1.9 Contact the Paducah BJC Sample Manager to develop the Statement of Work (SOW) for new activities or to notify of sample requests that are routine.

NOTE: Routine sampling activities (i.e., groundwater, environmental monitoring, Kentucky Pollutant Discharge Elimination System [KPDES], etc.) are reviewed on a periodic basis.

5.1.10 Ensure the SOW contains the required methods and deliverables.

Sample Personnel

5.1.11 Collect samples **and** forward them to the laboratory.

ACTION STEPS

5.2 Laboratory Contractual Screening

Paducah BJC Sample Manager

5.2.1 Upon receipt of data, conduct contractual screening to the SOW. (To the extent possible, perform contractual screening electronically.)

5.2.2 Identify **and** document any exceptions to the SOW.

Paducah BJC Sample Manager

5.2.3 Notify the requester and other data users (such as waste management coordinators) that the initial data has been received.

5.2.4 Provide a copy of the data deliverables (including the SOW exceptions) to the Paducah BJC Data Manager and the requester.

ACTION STEPS

5.3 Data Verification

Paducah BJC Data Manager and Requester

5.3.1 Perform electronic and/or manual review as defined in Step 5.1.8.

Requester

5.3.2 **IF** review will be performed manually, **THEN**
contact the Paducah BJC Data Manager for correct process on flagging protocol.

5.3.3 Flag data as necessary for explainable deviations.

5.3.4 **IF** electronic review is performed, **THEN**
ensure that data will be flagged by the Paducah BJC Data Manager.

5.3.5 **IF** the verification is done manually, **THEN**
provide flags to the Paducah BJC Data Manager for input into the data system.

Paducah BJC Sample Manager and Requester

5.3.6 Resolve issues identified during contractual screening or electronic data review with the laboratory.

5.3.7 **IF** data has been corrected, **THEN**

[a] Re-evaluate using the electronic data review process.

[b] Return to Step 5.3.1.

Requester

5.3.8 **IF** deviations in the data cannot be readily resolved with the samplers, laboratory, and requester, **THEN**

[a] Determine the usability of the data or the need for additional review of the data.

[b] Coordinate with the Paducah BJC Sample Manager to ensure that resources are identified for any additional work.

ACTION STEPS	5.4 Data Validation
	NOTE: Data validation must be accompanied by data assessment.
Requester	**5.4.1** Initiate data validation as defined in Step 5.1.6.
	5.4.2 **IF** validation is **NOT** required for the remaining data set, **THEN** GO TO Step 5.4.10.
Paducah BJC Sample Manager	**5.4.3** Develop an SOW for the validation activity.
	5.4.4 Submit the data packages to the validator or validation service selected.
	5.4.5 Upon receipt of the validation deliverables, conduct a contractual screening against the validation SOW.
Requester and Paducah BJC Quality	**5.4.6** Review the results of the validation process.
Paducah BJC Sample Manager	**5.4.7** **IF** validation or deliverables are **NOT** acceptable, **THEN** resolve discrepancies with validator or validation service until acceptable.
	5.4.8 Provide the Paducah BJC Data Manager with electronic <u>and</u> hard copies of the data validation deliverables.
Paducah BJC Data Manager	**5.4.9** Enter validation qualifiers into the data system.
	5.4.10 Notify the requester that data is complete and ready for review and assessment.
	5.4.11 Provide the requester with an electronic and hard copy of the data, quality checks, and validation results (if applicable) **OR** provide instructions to the requester on where to access the data.

ACTION STEPS	5.5 Data Assessment and Determination of Data Usability
	NOTE: Data validation can help ensure analyses are correct; however, data assessment must be performed to determine the data quality level (Data of Known Quality or Information Only Data) and to ensure data is useable.
Requester	**5.5.1** **WHEN** the data report is received from the Paducah BJC Data Manager, **THEN** conduct data assessment using the Attachment I, "Data Assessment Review Checklist."

Requester

5.5.2 Provide a completed Data Assessment Review Checklist to the Paducah BJC Data Manager.

Paducah BJC Data Manager

5.5.3 Make data available for final reporting.

Requester

5.5.4 Make program/project or activity decisions based on data.

5.5.5 **IF** additional data needs to be collected, **THEN**
return to Step 5.1.2.

5.5.6 Prepare final reports, as applicable, **and** submit to the Paducah BJC Records Manager.

ACTION STEPS

5.6 Data Release to External Agencies

Release Requester(s)

NOTE: An Authorized Derivative Classifier (ADC) review is requested to ensure that the data or document does not contain any classified information. A Technical Information Officer (TIO) review is required prior to release of documents and/or information (e.g., data) to any parties outside BJC, its subcontractors, and DOE.

5.6.1 Complete the Attachment J, "Paducah BJC Data Release to External Agencies" form, ensuring that all necessary reviews and approval signatures are collected prior to data release.

Paducah BJC Data Manager **or** Release Requester(s)

NOTE #1: **Electronic data formats** will contain a "read me" file that will identify the electronic data package and the number of files associated with the package. The "read me" file will also indicate the appropriate data qualifiers, along with their associated definitions and the appropriate data quality level.

NOTE #2: **Hard copy data formats** will contain a cover letter that will identify the contents of the data package. The cover letter will also indicate the appropriate data qualifiers, along with their associated definitions and the appropriate data quality level.

5.6.2 Transmit data, either hard copy or electronically, where applicable and requested.

Release Requester(s)

5.6.3 **IF** data is released by someone other than the Paducah BJC Data Manager, **THEN**
notify the Paducah BJC Data Manager of the requested transmittal.

ACTION STEPS

5.7 Records Management

Requester/ Release Requester

5.7.1 Ensure that all program/project records associated with the data collection activity, including all forms generated from this procedure, are transmitted to the Paducah BJC Document Management Center (DMC) for final disposition.

RECORDS

6.1 Records Generated

6.1.1 Analytical SOW

6.1.2 Data Assessment Review Checklist

6.1.3 Data packages

6.1.4 DQOs (meeting minutes, answers to Attachment D questions)

6.1.5 Data Quality Checks Form

6.1.6 Data Release to External Agencies Form

6.1.7 EMP

6.1.8 QAP

6.1.9 SAP

6.1.10 Validation SOW

6.1.11 WMP

6.2 Records Requirements

6.2.1 Generate **and** maintain records in accordance with requirements of BJC procedure BJC-OS-1001, *Records Management, Including Document Control.*

6.2.2 Submit records to the Document Management Center in accordance with requirements of Paducah BJC project procedure PA–1011, *Paducah Document Management Center – Access, Submittals, and Requests.*

SOURCE REFERENCES

7.1 KY/EM-174, *Safety Analysis Report-Paducah Gaseous Diffusion Plant, Paducah, Kentucky*

7.2 EPA 540-R-93-071, *Data Quality Objectives Process for Superfund, Interim Final Guidance*

7.3 EPA QA/G-9, *Guidance for Environmental Data Quality Assessment*

7.4 ES/ER/TM-88/R1, *Environmental Data Management Implementation Handbook for the Environmental Restoration Program*

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 4

Definitions

Contractual Screening - A process of evaluating a set of data against the requirements specified in the SOW to ensure that all requested information is received. The contractual screening includes, but is not limited to, the chain of custody (COC), number of samples, analytes requested, total number of analyses. method used, quality control samples analyzed, electronic data deliverables, units, holding times, and reporting limits achieved.

Data Assessment - A process for assuring that the type, quality, and quantity of data are appropriate for their intended use. It allows for the determination that the decision (or estimate) can be made with the desired level of confidence, given the quality of the data set. Data assessment follows Data Verification and can be performed in parallel with Data Validation. Data assessment must be performed to ensure data is useable.

Data Manager - Person responsible for long-term electronic storage of data, for loading electronic data deliverables (EDD), performing electronic verification of data, and ensuring compliance to the Paducah BJC Data Management Policy.

Data of Known Quality - Data, along with appropriate laboratory, verification, validation, and assessment qualifiers, that can be used for decision making purposes and was collected and managed according to this procedure.

Data Quality Checks - A list of quality control elements associated with a data collection activity which are evaluated during data verification and/or data validation.

Data Quality Level - A description that indicates whether the data was generated according to this procedure. The two levels are Data of Known Quality and Information Only Data.

Data Quality Objectives - A set of criteria established for the collection of data. The DQO process is a planning tool based on the scientific method that clearly identifies an environmental problem; the remedial decisions to address the problem; and the type, quantity, and quality of data needed to support the decision. This process is based on the DQO process developed by the Environmental Protection Agency (EPA). The DQO process may be applied in modified form to any data collection activity. The DQO process balances risk with cost in selecting the most appropriate data collection plan.

Data Validation - A process performed for a data set by a qualified individual independent from sampling, laboratory, program/project management, or other decision making personnel for the program/project. Data validation evaluates the laboratory adherence to analytical method requirements.

Data Verification - A process for comparing a data set against a set standard or contractual requirement. Verification may be performed electronically, manually, or by a combination of both. Data verification includes contractual screening and can include other data quality checks established by the program/project team.

Attachment A
DEFINITIONS/ACRONYMS
Page 2 of 4

Definitive Data - Analytical measurements for which the presence, and corresponding concentration, of the target analyte(s) can be determined with a known degree of certainty. The measurements are supported with appropriate physical evidence documenting the acquisition and analysis. Definitive data in electronic form must be supported with retrievable, but not necessarily retrieved, physical evidence in the laboratory. This evidence can include analytical results, QA/QC results, chain of custody, logbooks, standards information, etc.

Electronic Data Deliverables - Data that is received in electronic format, either through transfer on physical media or direct communication between computerized data management systems. EDD contents must meet defined completeness, consistency, and format requirements. These criteria are defined in the SOW for each program or project.

External Agency - Any organization external to BJC, its subcontractors, and DOE.

Historical Data - Data that was collected and managed prior to implementation of this procedure.

Information Only Data - Data for which quality is not assured and may or may not contain the appropriate qualifiers; however, data can be used for informational purposes or can be used for decision making with relevant documentation.

PARCC Parameters - Precision, Accuracy, Representativeness, Completeness, and Comparability, as explained in Attachment E.

Program/Project Team - Personnel involved in the data collection planning, review, and decision making, and include, but are not limited to, the Requester, Quality Representative, Sample Manager, and the Data Manager.

Quality Assured Data - Data that has undergone a documented review, as specified by this procedure, to provide adequate confidence that the data conforms to established technical requirements and is sufficient for the intended use.

Release Requester - Person who requests release of data to an external agency.

Requester - A project manager (or his designated representative, such as a technical lead, risk assessor, waste management coordinator, compliance coordinator, or other individual who determines the need for data to support decision making. The requester is responsible for coordinating sample collection, sample analysis, data assessment, and decision making. If the requestor is a designated representative, the project manager has ultimate responsibility.

Attachment A
DEFINITIONS/ACRONYMS
Page 3 of 4

Sample Manager - Person responsible for interfacing with the Paducah BJC Sample Management Office for procurement of laboratory services and/or validation services and coordinate contractual screening.

Screening Data - Measurements generated through the use of field or fixed laboratory methods in which the level of certainty in the data cannot be determined given physical evidence documenting the acquisition and analysis of the sample. Analytical methods producing field measurements or screening quality data include those that indicate the presence or absence of an analyte or class of analytes, or provide a semi-quantitative (estimated) result. Field measurement and other screening quality data include, but are not limited to, Draeger tube; organic vapor analysis (OVA); soil gas surveys; radiation and contamination monitoring; and measurements for pH, conductivity, temperature, dissolved oxygen, and turbidity. Screening data results may be confirmed by collecting a specified percentage of definitive data.

Statement of Work - The contractual agreement between the requesting organization and the service provider. The SOW defines the scope of work including associated QA/QC, schedules, and deliverables.

Acronyms

ADC - Authorized Derivative Classifier
ASTM - American Society for Testing Materials
BJC – Bechtel Jacobs Company LLC
COC - Chain of Custody
DOE - United States Department of Energy
DMC - Document Management Center
DQOs - Data Quality Objectives
EDD - Electronic Data Deliverables
EMP - Environmental Monitoring Plan
EPA - United States Environmental Protection Agency
ISMS – Integrated Safety Management System
KPDES - Kentucky Pollutant Discharge Elimination System
MS - Matrix Spike
MSD - Matrix Spike Duplicate
OVA - Organic Vapor Analysis
PARCC - Precision, Accuracy, Representativeness, Completeness, and Comparability
PCB - polychlorinated biphenyl
PGDP - Paducah Gaseous Diffusion Plant
pH – Hydrogen ion potential
QAP - Quality Assurance Plan
QA - Quality Assurance
QC - Quality Control

Attachment A
DEFINITIONS/ACRONYMS
Page 4 of 4

RI/FS - Remedial Investigation/Feasibility Study
SAP - Sampling and Analysis Plan
SOW - Statement of Work
STR – Subcontract Technical Representative
TIO - Technical Information Officer
TSD - Treatment Storage Disposal
VOA - Volatile Organic Analysis
WAC - Waste Acceptance Criteria
WM - Waste Management
WMP - Waste Management Plan

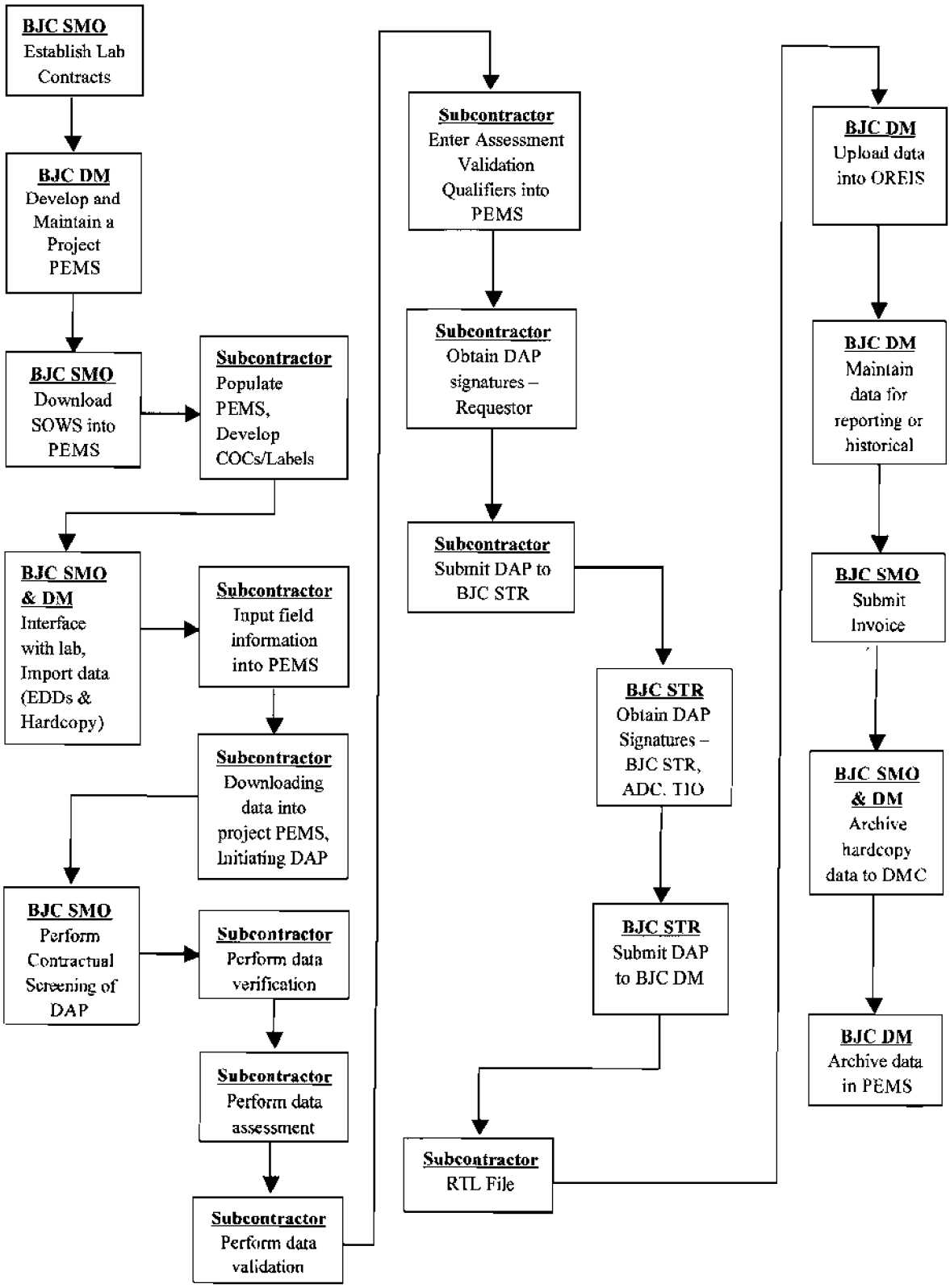
NOTE: The above includes all acronyms used in the text of this procedure and in the other attachments.

Attachment B
PADUCAH SAMPLING AND DATA MANAGEMENT FLOWCHART
Page 1 of 2

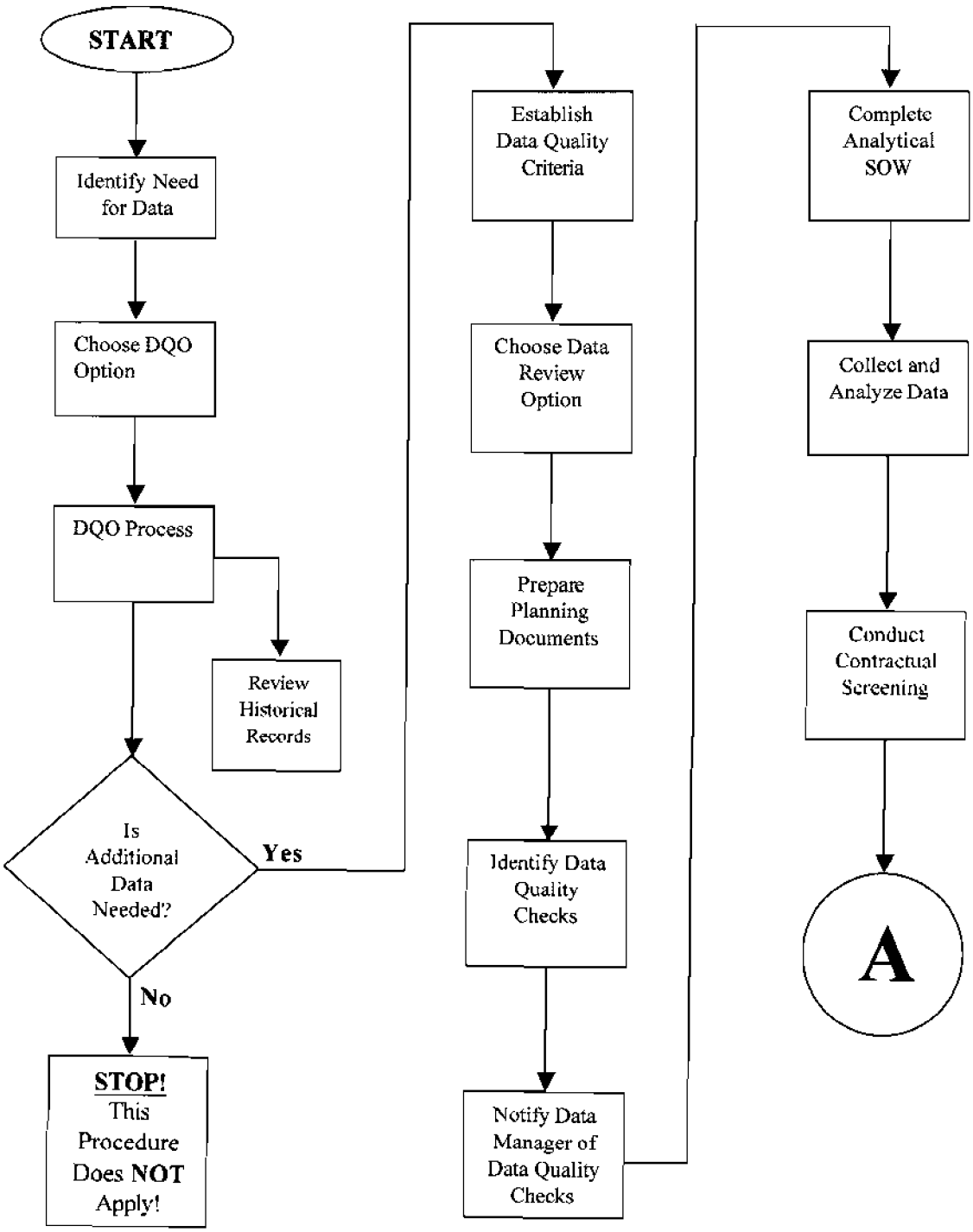
The following acronyms are used in the flowchart on the next page:

ADC	Authorized Derivative Classifier
BJC	Bechtel Jacobs Company LLC
COC	Chain of Custody
DAP	Data Assessment Package
DM	Data Management
DMC	Document Management Center
DOE	Department of Energy
EDD	Electronic Data Deliverable
OREIS	Oak Ridge Environmental Information System
PEMS	Project Environmental Measurements System
RTL	Ready to Load
SMO	Sample Management Office
SOW	Statement of Work
STR	Subcontract Technical Representative
TIO	Technical Information Officer

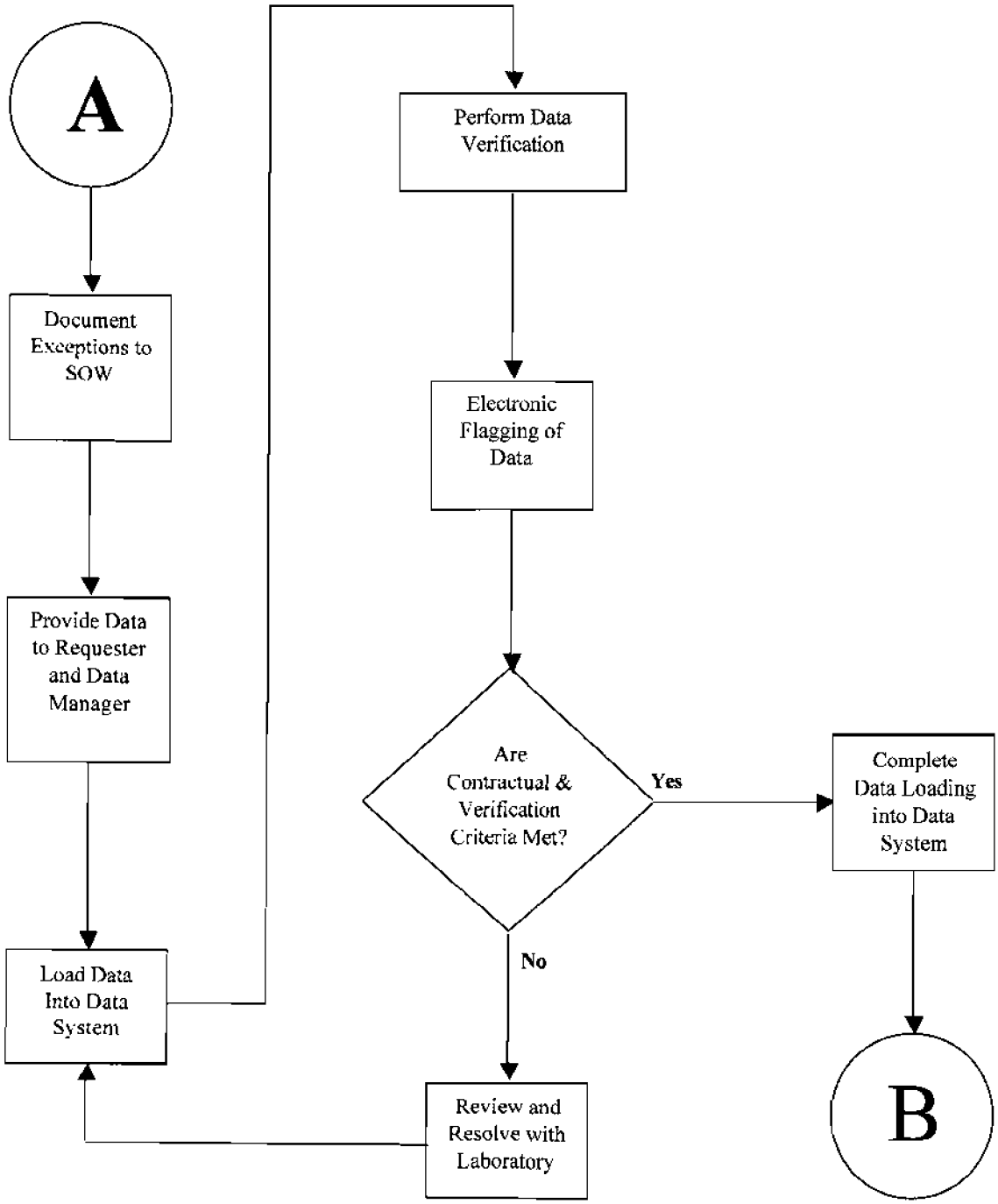
Attachment B - Paducah Sampling and Data Management Flowchart – Page 2 of 2



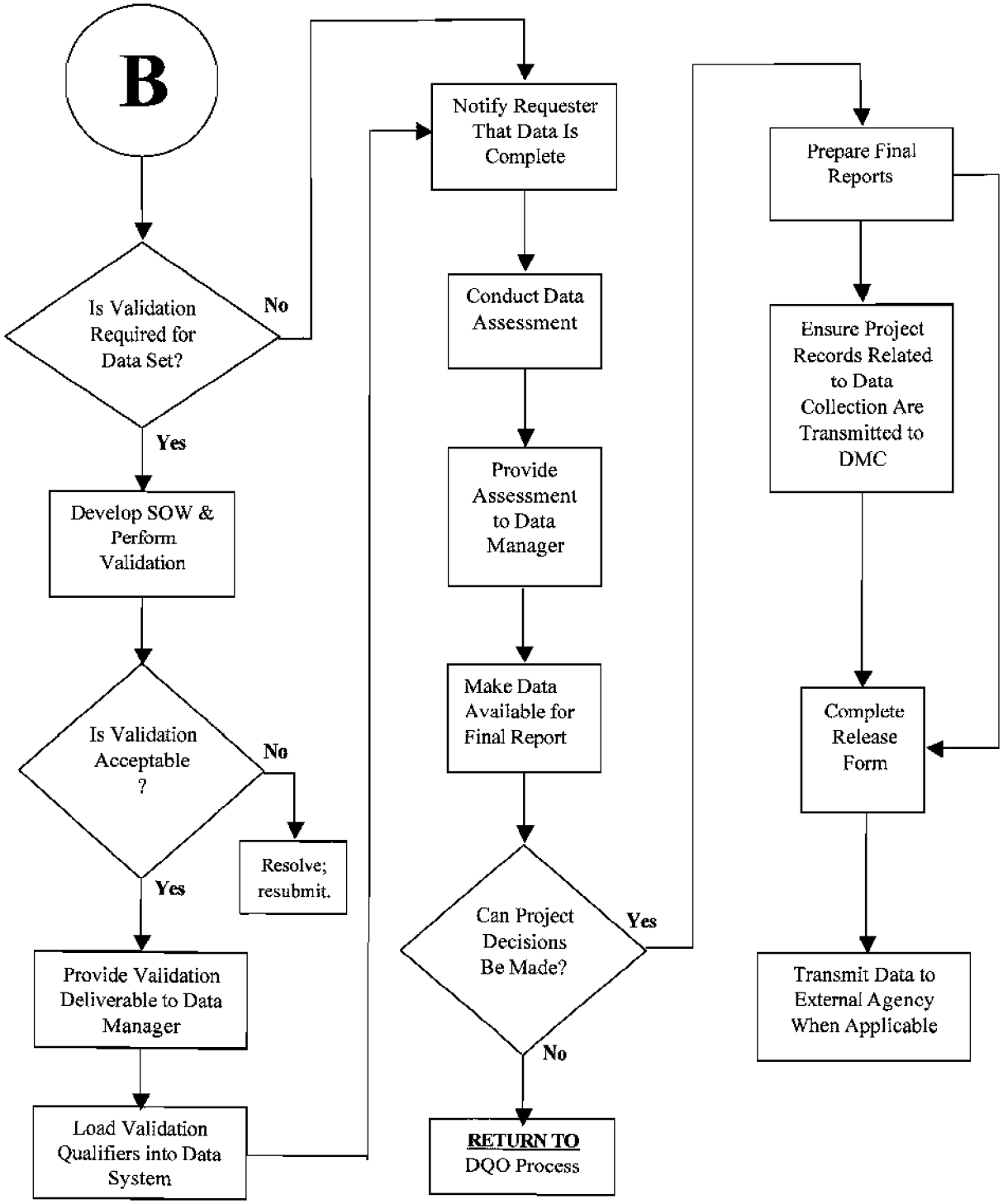
Attachment C
PADUCAH BJC DATA CYCLE FLOWCHART
Page 1 of 3



Attachment C
PADUCAH BJC DATA CYCLE FLOWCHART
Page 2 of 3



Attachment C
PADUCAH BJC DATA CYCLE FLOWCHART
Page 3 of 3



Attachment D
OPTIONS TO IMPLEMENTING AND DOCUMENTING THE DQO PROCESS FOR PADUCAH BJC PROGRAMS/PROJECTS

INTRODUCTION

The DQO Process is a scientific and legally-defensible data collection and planning process to help users decide what type, quality, and quantity of data will be sufficient for decision making. This attachment is based on a series of planning steps designed to assure that data collected is adequate for the intended purpose.

PURPOSE

The purpose of this attachment is to provide options for implementing and documenting the DQO process.

DQO OPTIONS AND APPLICABILITY

Option 1

For ER programs/projects, the detailed approach as found in the EPA Data Quality Objectives Process for Superfund and its implementing procedure, BJC-ES-1004 (ES-A-0101), *Implementing and Documenting the DQO Process for Environmental Restoration Projects*, is appropriate. For long-term environmental monitoring sampling programs and extensive waste sampling activities, this detailed and structured approach can be useful. However, full implementation of the process may not always be appropriate.

Option 2 (Minimum Requirements)

The following tables are provided for guidance in documenting a simplified version of the DOQ process. Use the applicable table for your program/project.

Table C.1 - ENVIRONMENTAL MONITORING PROGRAMS/PROJECTS - DQO PROCESS

Table C.2 - ENVIRONMENTAL RESTORATION PROGRAMS/PROJECTS - DQO PROCESS

Table C.3 - SITE CHARACTERIZATION PROGRAMS/PROJECTS - DQO PROCESS

Table C.4 - WASTE CHARACTERIZATION PROGRAMS/PROJECTS - DQO PROCESS

Attachment D
OPTIONS TO IMPLEMENTING AND DOCUMENTING THE DQO PROCESS FOR PADUCAH BJC PROGRAMS/PROJECTS
Page 2 of 6

Option 3

A user-defined DQO process that includes the minimum requirements from Option 2 and any additional actions needed.

APPLICABILITY EXCLUSIONS

This attachment is **not** applicable to the Environmental Monitoring Program's PCB spills, asbestos events, and environmental spills due to the quick response time and the well-defined actions to be taken in the event of the occurrence.

DOCUMENTATION

Documentation of the DQO Process is required and will:

- provide a source of historic data and process knowledge for related sampling,
- provide a tool for conducting data assessment
- facilitate efficient program/project management transfers, or
- allow decisions to be recalled and defended.

The documentation may be presented in various ways and will include:

- an outline or text form following the format shown in this attachment. Include responses to the questions as separate, brief accounts of the information gathered, its sources, and the rationale for decisions made.
- references to various other documents, such as SAPs, QAPs, EMPs, WMPs, Data Management Plans for ER programs/projects, etc., as necessary.

Attachment D - Page 3 of 6
Table C.1 - ENVIRONMENTAL MONITORING PROGRAMS/PROJECTS - DQO PROCESS

1. **The Problem and the Decision**--(The drivers for data collection activities.)
   - What is the description of the area of concern? (Where is the current location?)
   - What are the contaminants or analytes of interest? (What is the media of concern? What are the suspected contaminants? How were they selected? What are the known or potential routes of migration? What are the known or potential human and environmental receptors? What are the exposure pathways?)
   - What decision needs to be made regarding the area (i.e., disposition of waste, etc.)?

2. **Inputs to the Decision**--(The sources of data and information used to make the decision.)
   - What historical data exists? Is it adequate for use?
   - What process knowledge exists? Is it adequate for use?
   - What additional data must be collected? (What are the analytes and analytical methods?)

3. **Physical Boundaries to be Considered**--(Physical characteristics that affect the sampling design.)
   - What is the location of the potential contamination? (What are the depth and boundaries/geometry of the potential contamination area?)
   - What considerations affect the sample location choices? (Is the intention to characterize the average of the environmental media? What are the site conditions that affect sampling [i.e., power lines, trees, concrete pad, etc.]? Is it homogenous? Is the contamination level expected to be a continuous range?)
   - Are there other sampling constraints, such as temporal, schedule, seasonal concerns, regulatory requirements, etc.?

4. **Decision Statement and Uncertainty**
   - What are the steps to be taken after the analytical results are received? (Is this preliminary sampling? What results will trigger further testing, verification, or action? What additional steps will be taken? If I find ____, then what will I do? Consider the potential impact for making an incorrect decision based on the data.)

5. **Develop the Data Sampling Design**
   - State the type of data to be obtained. (Will it be screening, definitive, or a combination?)
   - State the approach to sample selection. (Will it be grab or composite, judgmental [selective] or random? Will it be a statistically-based selection?)
   - Optimize the design and approach for efficiency and effectiveness. (What confidence intervals are needed? What QA/QC will be required by sample method or this procedure? For minimum quality criteria, see Attachment E. For data validation requirements, see Attachment F. What additional QA/QC is requested?)

Attachment D - Page 4 of 6

Table C.2 - ENVIRONMENTAL RESTORATION PROGRAMS/PROJECTS - DQO PROCESS

1. **The Problem and the Decision**--(The drivers for data collection activities.)
   - What is the description of the area of concern? (Where is the current location?)
   - What are the contaminants or analyte of interest? (What is the media of concern? What are the suspected contaminants? How were they selected? What are the known or potential routes of migration? What are the known or potential human and environmental receptors? What are the exposure pathways?)
   - What are potential corrective actions for this problem?
   - What decision needs to be made regarding the area (i.e., disposition of waste, etc.)?

2. **Inputs to the Decision**--(The sources of the data and information used to make the decision.)
   - What historical data exists? Is it adequate for use?
   - What process knowledge exists? Is it adequate for use?
   - What additional data must be collected? (What are the analytes and analytical methods?)

3. **Physical Boundaries to be Considered**-- (Physical characteristics that affect the sampling design.)
   - What is the location of the potential contamination? (What are the depth and boundaries/geometry of the potential contamination area?)
   - What considerations affect the sample location choices? (Is the intention to characterize the average of the environmental media or do you need to know the "hot spots?" What are the site conditions that affect sampling [i.e., power lines, trees, concrete pad, etc.?] Is it homogenous? Is the contamination level expected to be a continuous range?)
   - Are there other sampling constraints such as temporal, schedule, seasonal concerns, regulatory requirements, etc.?

4. **Decision Statement and Uncertainty**
   - What are the steps to be taken after the analytical results are received? (Is this preliminary sampling? What results will trigger further testing, verification, or action? What additional steps will be taken? If I find _____, then what will I do? Consider the potential impact for making an incorrect decision based on the data.)

5. **Develop the Data Sampling Design**
   - State the type of data to be obtained. (Will it be screening, definitive, or a combination?)
   - State the approach to sample selection. (Will it be grab or composite, judgmental [selective] or random? Will it be a statistically-based selection?)
   - Optimize the design and approach for efficiency and effectiveness. (What confidence intervals are needed? What QA/QC will be required by sample method or this procedure? For minimum quality criteria, see Attachment E. For data validation requirements, see Attachment F. What additional QA/QC is requested?)

Attachment D - Page 5 of 6

Table C.3 - SITE CHARACTERIZATION PROGRAMS/PROJECTS - DQO PROCESS

1. **The Problem and the Decision**--(The drivers for data collection activities.)
   - What is the description of the area of concern? (Where is the location?)
   - What are the boundaries of the area that will be characterized?
   - What are the contaminants or analytes of interest? (What is the media of concern? What are the suspected contaminants? How were they selected?)

2. **Inputs to the Decision**--(The sources of the data and information used to make the decision.)
   - What historical data exists? Is it adequate for use?
   - What process knowledge exists? Is it adequate for use?
   - What additional data must be collected? (What are the analytes and analytical methods?)

3. **Physical Boundaries to be Considered**--(Physical characteristics that affect the sampling design.)
   - What is the location of the potential contamination? (What are the depth and boundaries/geometry of the potential contamination area?)
   - What considerations affect the sample location choices? (Is the intention to characterize the average of the environmental media? What are the site conditions that affect sampling [i.e., power lines, trees, concrete pad, etc.?] Is it homogenous? Is the contamination level expected to be a continuous range?)
   - Are there other sampling constraints, such as temporal, schedule, seasonal concerns, regulatory requirements, etc.?

4. **Decision Statement and Uncertainty**
   - What are the steps to be taken after the analytical results are received? (Is this preliminary sampling? For what event? What results will trigger further testing , verification, or action? What additional steps will be taken? If I find _____, then what will I do? Consider the potential impact for making an incorrect decision based on the data.)

5. **Develop the Data Sampling Design**
   - State the type of data to be obtained. (Will it be screening, definitive, or a combination?)
   - State the approach to sample selection. (Will it be grab or composite, judgmental [selective] or random? Will it be a statistically-based selection?)
   - Optimize the design and approach for efficiency and effectiveness. (What confidence intervals are needed? What QA/QC will be required by sample method or this procedure? For minimum quality criteria, see Attachment E. For data validation requirements, see Attachment F. What additional QA/QC is requested?)

Attachment D - Page 6 of 6

Table C.4 - WASTE CHARACTERIZATION PROGRAMS/PROJECTS - DQO PROCESS

1. **The Problem and the Decision**--(The drivers for data collection activities.)
   - What is the description of the waste? (Where and when was it generated? What is the media and the volume? Where is it now?)
   - Who needs information about the waste? Why do they need the information? (WM for characterization purposes? WM to determine TSD options? WM to meet a specific vendor's WAC?)
   - What are the contaminants or analytes of interest? (What are the suspected contaminants? How were they selected?)
   - What decision needs to be made regarding the area (i.e., disposition of waste, etc.)?

2. **Inputs to the Decision**--(The sources of the data and information used to make the decision.)
   - What historical data exists? Is it adequate for use?
   - What additional data must be collected? (What are the analytes and analytical methods?)

3. **Physical Boundaries to be Considered**--(Physical characteristics of waste that affect sampling design.)
   - What is the location of the potential contamination? (Surface contamination or volumetric?)
   - What considerations affect the sample location choices? (Is the intention to characterize the average of the waste stream or do you need to know the "hot spots?")
   - How is the waste containerized?
   - Are there sampling problems? (What is the geometry of the waste? Is it homogenous? Is the contamination level expected to be a continuous range?)
   - Are there other sampling constraints, such as temporal, schedule, seasonal concerns, regulatory requirements, etc.?

4. **Decision Statement and Uncertainty**
   - What are the steps to be taken after the analytical results are received? (Is this preliminary sampling? What results will trigger further testing, verification, or action? What additional steps will be taken? If I find _____, then what will I do? Consider the potential liability for making an incorrect characterization decision based on the data.)

5. **Develop the Data Sampling Design**
   - State the type of data to be obtained. (Will it be screening, definitive, or a combination?)
   - State the approach to sample selection. (Will it be grab or composite, judgmental [selective] or random? Will it be a statistically-based selection?)
   - Optimize the design and approach for efficiency and effectiveness. (What confidence intervals are needed? What QA/QC will be required by sample method or this procedure? For minimum quality criteria, see Attachment E. For data validation requirements, see Attachment F. What additional QA/QC is requested?)

Attachment E
PADUCAH BJC DATA QUALITY REFERENCE LIST

INTRODUCTION

The following information is an aid to the program/project manager, program/project scoping team members, and/or DQO facilitators to select the program/project data quality elements. This information should be obtained during the sampling design optimization step in Attachment D, Step 5, or the DQO Process, Step 7. The minimum requirements are listed for screening and/or definitive data. A program/project manager may choose to implement quality control above the minimum requirements; however, certain data quality elements are not applicable to screening data.

PURPOSE

The purpose of this attachment is to provide a reference list of data quality elements and data quality requirements for a data collection activity. The selected elements should be incorporated into applicable program/project plans.

SCREENING AND DEFINITIVE DATA

There are two types of data generated using this procedure. "Screening Data" is defined in Attachment A and generally refers to qualitative data. Screening data has been previously termed EPA Levels I and II. In order to increase confidence, screening data results should be confirmed by collecting a specified percentage of definitive data. **The recommended percentage of definitive data for confirming screening data is 10 percent.** This, in turn, makes the data more usable for decision making. "Definitive Data" is also defined in Attachment A and describes data usually generated from a fixed-based laboratory following appropriate quality control requirements for various analytical methods.

Definitive data has been previously termed EPA Levels III, IV and V. In this attachment and in Attachment F, screening data is categorized by S, S1, or S2, depending on the level of detail needed for the data collection activity. Definitive data is categorized by D, D3A, D3B, D4, and D5. Attachment F provides additional explanation and examples for the categories.

Attachment E
PADUCAH BJC DATA QUALITY REFERENCE LIST
Page 2 of 4

PARCC PARAMETERS

Data are only useable if the precision and accuracy is known. Data is only useable for decision making if it is also precise, accurate, representative of the whole, comparable to expectations, and complete as planned. These requirements are known as the PARCC parameters and are explained in detail below. Data quality criteria should be chosen to address all five parameters. The PARCC parameters should be reviewed during data assessment, as defined later in Attachment I.

Precision--a quantitative measurement of the variability of a group of measurements as compared to their average. Usually expressed as a percentage or a standard deviation, it evaluates the reproducibility of the system. Sample duplicates measure the reproducibility of the sampling event, while lab replicates measure the precision of the analytical process. The acceptable precision may be defined by the laboratory method used.

Accuracy--a quantitative measurement of the bias of the data. It represents how close the measurement data is to the true value. Sampling accuracy can be assessed by evaluating field and trip blanks. Analytical accuracy is measured by percent recoveries associated with the laboratory analytical control spikes (blank spikes), surrogate spikes, or matrix spikes. The acceptable accuracy may be defined by the laboratory method used.

Representativeness--a qualitative measurement of the ability of a sample or group of data to adequately describe or define the conditions being measured. Precision, accuracy, and completeness all affect representativeness. Sampling strategy (location, method, and frequency) is critical to assure that the samples statistically represent the population. Laboratory precision and accuracy reflect how representative the data is of the sample.

Completeness--a quantitative measurement of the percentage of acceptable data as compared to the number planned. Both sampling and analytical completeness can be measured.

Comparability - a qualitative measurement of the confidence with which one data set can be compared with another. Comparability is achieved by using standard techniques for collection and analysis.

Attachment E - Page 3 of 4

PADUCAH BJC DATA QUALITY REFERENCE LIST		
Data Quality Element	**Minimum For: Screening (S) Definitive (D)**	**PARCC**
Field Sampling Quality Control		
Sample Logbooks	S, D	Representativeness, Completeness
Sample Chain of Custody (COC)	S, D	Representativeness, Completeness
Transcription - Logbook vs. COC	S, D	Representativeness, Completeness
Containers	S, D	Representativeness
Preservation	S, D	Representativeness
Field Duplicates	S, D (5% Min for S, D)	Precision
Trip Blanks (VOA Only)	S, D (5% Min for S, D)	Accuracy
Field Blanks	S, D (5% Min for S, D)	Accuracy
Equipment Rinseates	S, D (5% Min for S, D)	Accuracy
Sampling Completeness Requirement		Representativeness, Completeness
Field/Laboratory Methods [a]	**Screening:** Analyte or instrument specific **Definitive:** SW-846, EPA, ASTM	
Analytical/Measurement Quality Control [b]		
Initial Calibration of Instrument	S, D	Accuracy
Calibration Check of Instrument	S, D	Accuracy
Calibration Range		Accuracy
Reporting Detection Limits (Method)	S, D	Comparability
Analytical Error Determination		Precision, Accuracy
Laboratory COC		Representativeness, Completeness
Transcription COC vs Samples		Representativeness, Completeness
Holding Times	S, D	Representativeness, Comparability
Analytical Method	S, D	Comparability
Method Units	S, D	Accuracy, Comparability
Calculation Verifications		Representativeness, Completeness
Transcription-Lab data vs. EDD/Report		Representativeness, Completeness
Analytical Completeness Requirement		Precision
Lab Duplicates	S^b, D^b	Accuracy, Precision
Blank Duplicates		Accuracy
Reagent Blanks	D^b	Accuracy
Method Blanks	D^b	Accuracy
Spikes/Laboratory Control Samples	D^b	Accuracy
Matrix Spikes	D^b	Precision
Matrix Spike Duplicates	D^b	Accuracy
Post Digestion Spikes	D^b	Precision, Accuracy
Performance Samples	S^b, D^b	Accuracy
Interference Check Samples	D^b	Accuracy

Attachment E - Page 4 of 4

PADUCAH BJC DATA QUALITY REFERENCE LIST		
Data Quality Element	**Minimum for: Screening (S) Definitive (D)**	**PARCC**
Analytical Deliverables	Electronic Data Deliverables (EDD) and hard copy results	
Identification number (sample number or location name)	S, D	
Date/time sampled	S, D	
Lab sample number	D	
Date analyzed	S, D	
Date completed	S2, D	
Parameter/analyte	S, D	
Qualifier	S, D	
Results	S, D	
Units	S, D	
Comments	S2, D	
Method (lab and field)	S2, D	
Blanks	S2, D	
Spikes (MS*, MSD*, blank [DI water spiked--provides feedback on the matrix effect])	D	
Surrogates, if applicable	D	
Lab duplicates*	D	
Reporting Detection Limits	D	
Former Level III Data Package Former Level IV Data Package Former Level V Data Package	These data packages include minimum definitive data elements plus additional information.	
Data Verification Percentages	S, D 100% for both	
Data Validation Percentages	D 5% Min**	
Data Assessment[c]	100%	

[a] If ER, waste characterization, or compliance monitoring activities are planned, SW-846 methods must be considered. If SW-846 methods are not available, use EPA-approved methods. If a remedial design is planned, ASTM methods must be considered. If environmental monitoring data is collected, EPA methods must be considered.

[b] Analytical quality control is dependent on the method specified.

[c] NOTE: 100% of the data should be assessed. However, individual project records, such as logbooks, chain of custody forms, etc., should be reviewed on a program/project designated frequency.

* Lab duplicates are optional and can be performed at lab or customer request. If doing a field duplicate, a lab duplicate is not value added.

** A greater percentage of validation may be required for some programs/projects (i.e., risk assessments and remedial investigations). The program/project teams can increase as needed to ensure valid data.

S = S1 or S2 as defined in Attachment F.

D = D3A, D3B, D4, or D5 as defined in Attachment F.

Attachment F
OPTIONS FOR DATA REVIEW
Page 1 of 3

INTRODUCTION

To ensure the process for data quality continues, data review must be performed for results received from a data collection activity. The three elements of data review outlined in this procedure are verification, validation, and assessment.

PURPOSE

The purpose of this attachment is to provide guidelines for data review. The documentation checklist to be used for assessment of a data collection activity is also provided in this attachment.

DATA VERIFICATION

Data verification is the first step of data review. The preferred method for performing verification is electronic. Verification criteria are selected in Section 4.1.5 of this procedure and documented using Attachment H. The extent of verification is based on the data category as demonstrated in the table below.

DATA VALIDATION

Data validation follows verification in the data review process. The data validation options in this attachment are similar to the format specified by the former EPA data quality levels with the exception of diverging from the former EPA Level III data validation. Grade 3A, as listed in the following Review Options and Applicability table, is a less rigorous form of validation based on the minimum data deliverable requirements. Grade 3B, Grade 4, and Grade 5 are the same as the former EPA Level III, Level IV, and Level V data validation, respectively. All grades of validation must be performed by a third party. Third party validation is defined as validation performed by persons independent from sampling, laboratory, and decision making for the program/project (i.e., not the program/project manager). Data validation is documented in a formal deliverable from the data validator.

The option chosen (level and frequency) for validation is based on data category and the following considerations:

- Regulatory drivers/requirements
- End-user of data
- Future applicability of the data (other users such as regulatory agencies, risk assessment personnel, internal users, etc.)
- Legal ramifications and defensibility of data
- Confidence in laboratory

The data set to be validated may be determined programmatically or by the individual program/project. The option chosen for data validation should be made by the program/project team.

Attachment F
OPTIONS FOR DATA REVIEW
Page 2 of 3

DATA ASSESSMENT

Data assessment is the last review step prior to release of the data from the program/project team. It is an integration of all information collected about a result. Data verification and validation can ensure analyses are correct; however, data assessment must be performed to evaluate data usability. This includes a review of the data itself, the results of all previous reviews of the data, and evaluation against the intended purpose for data collected. Data assessment must be performed for all data collection activities and documented using the Data Assessment Review Checklist in Attachment I. Data assessment is required prior to use of the data, or data release into the final data repository. Data assessment frequency is determined based on decision making and releasibility requirements. This decision is made by the program/project team.

Attachment F - Page 3 of 3

REVIEW OPTIONS AND APPLICABILITY

Data Category	Examples for Generation of Category	Former Level of Data	Data Verification	Data Validation	Data Assessment
Screening Data S1	OVA Qualitative	Level I or A	100% Grade 1 or None Review only the sample results presented.	NA	100%
Screening Data S2	Portable field GC Hydrolab pH, Conductivity Qualitative Semiquantitative	Level II or B	100% Grade 2 Electronic review of data. Review of quality control samples as defined in Attachment H.	NA	100% Comparison to definitive data results, if applicable.
Definitive Data D3A	Routine laboratory Quantitative	Level III or C	100% Grade 3A Validation would consist of looking at the criteria in the minimum lab deliverable in Attachment E, plus any additional information required for the program/project.	5%	100%
Definative Data D3B	Routine laboratory RI/FS Quantitative	Level III or C	100% Grade 3B Traditional Level III data validation on a data package.	5%	100%
Definitive Data D4	Routine laboratory Quantitative, RI/FS More rigorous QC	Level IV or D	100% Grade 4 Same as Grade 3B plus raw data.	5%	100%
Definitive Data D5	Not standard methods Unusual parameters	Level V or E	100% Grade 5 Same as Grade 3B on the user-defined lab deliverable.	5%	100%

Attachment G
ESTIMATED TIMEFRAME FOR ROUTINE DATA REVIEW PROCESS
Page 1 of 1

Item #	Activity	Timeframe*
1	Sample Laboratory Turnaround (from sample collection)	30-45 days
2	Data Verification	1-5 working days
3	Data Validation	30-45 days
4	Data Assessment	10-15 days

The above estimated timeframe for data review is listed in order of occurrence and the steps are sequential. Once the final step of the data review process (data assessment) has been completed, the data is ready for risk assessment/ report writing. It should be mentioned that utilization of the data prior to completion of the data review process might result in drawing conclusions or making decisions on incorrect or unusable data. This in turn can result in added cost and duplication of effort.

* The steps listed in this table can be expedited based upon project needs and the projects ability to pay the extra cost required to expedite the work. In addition, if the process identifies any problems, the timeframe may require extension.

Attachment H
IDENTIFICATION OF PROGRAM/PROJECT DATA QUALITY CHECKS
Page 1 of 3

INTRODUCTION

The Data Quality Checks for the program/project are identified in the reference list in Attachment E. This checklist will be used by applicable data review personnel to perform verification and some aspects of validation and assessment.

PURPOSE

The purpose of this attachment is to document the selection of program/project-specific data quality checks, to define the review methods, and to indicate the personnel responsible for performing the review.

INSTRUCTIONS FOR COMPLETING THIS ATTACHMENT

The **Data Review Step** column provides the data review step in which the individual QC element is reviewed. The verification, validation, and assessment frequencies of the QC elements identified in the table should be performed according to the minimum requirements specified in Attachment E. The assessment of the QC element during the life cycle of the data collection activity will be performed in one (or a combination) of the following ways: 1) by QA during program/project surveillances, 2) by the project team by defined activities, or 3) by the project manager during the completion of Attachment I. After the data quality checks for the program/project have been identified, the **Applicable** column should be completed. Please ensure that the minimum requirements listed in Attachment E are identified for the data type, and checked appropriately. Mark the **Applicable** column for the quality check with an "NA" if the check is not to be performed during the program/project and provide comments. The **Evaluation Method** describes how the check will be accomplished (i.e., manual or electronic) and lists the individual responsible for performing the functions. This form should be provided to the data review personnel. The **Completed By** should be signed upon completion of the review, and any comments relevant to the activity should be listed in the **Comments** column. The checklist should be filed in the program/project files.

The two-page Data Quality Check form is available on the next two pages without page headers.

DATA QUALITY CHECKS
(Page 1 of 2)

Program/Project Number: _____

Data Type--(Screening or Definitive): _____

QC Element	Data Review Step	Applicable	Evaluation Method	Completed By	Comments
FIELD SAMPLING QUALITY CONTROL					
Sample Logbooks	Assessment				
Sample Chain of Custody (COC)	Assessment				
Transcription--Logbook vs. COC	Assessment				
Containers	Assessment				
Preservation	Assessment				
Field Duplicates	Assessment				
Trip Blanks (VOA Only)	Assessment				
Field Blanks	Assessment				
Equipment Rinsates	Assessment				
Sampling Completeness	Assessment				
ANALYTICAL/MEASUREMENT QUALITY CONTROL					
Initial Calibration of Instrument	Validation				
Calibration Check of Instrument	Validation				
Calibration Range	Validation				
Reporting Detection Limits (Method)	Verification		Electronic Sample Manager		
Analytical Error Determination	Validation				
Laboratory COC	Validation				
Transcription COC vs. Samples	Validation				
Holding Times	Verification		Electronic Data Manager		

DATA QUALITY CHECKS
(Page 2 of 2)

Program/Project Number: _____

Data Type--(Screening or Definitive): _____

QC Element	Data Review Step	Applicable	Evaluation Method	Completed By	Comments
ANALYTICAL/MEASUREMENT QUALITY CONTROL (Continued)					
Analytical Method	Verification		Electronic Sample Manager		
Method Units	Verification		Electronic Data Manager		
Calculations	Validation				
Transcription--Lab data vs. EDD and Report	Validation				
Analytical Completeness	Verification		Electronic Sample Manager		
Lab Duplicates	Validation				
Blank Duplicates	Validation				
Reagent Blanks	Validation				
Method Blanks	Validation				
Spikes/Laboratory Control Samples	Validation				
Matrix Spikes	Validation				
Matrix Spike Duplicates	Validation				
Postdigestion Spikes	Validation				
Performance Samples	Validation				
Interference Check Samples	Validation				
OTHER CHECKING CRITERIA					

Attachment I
DATA ASSESSMENT REVIEW CHECKLIST
Page 1 of 2

A one-page form is available on the next page without page headers.

DATA ASSESSMENT REVIEW CHECKLIST

Program/Project Number:	Program/Project Title:			
Requester:				
ITEM	**YES**	**NO**	**NA**	**COMMENTS**
PLANNING Were the data quality objectives and sampling design sufficient to provide enough information for the current decision?				
DATA REVIEW Is data complete as planned?				
Have comments and/or data qualifiers from field and laboratory technicians and supporting information been considered (Data Qualifiers and Variances)?				
Are duplicates and quality control sample results acceptable (Spikes, Blanks, etc.)?				
Does data validation indicate that data is useable?				
Have the impacts of holding time violations been evaluated?				
Are analytical detection limits below or at regulatory or action limits?				
Is data reasonable when compared to known or expected parameters levels?				
Based upon previous data reviews and results of the above questions, is data of adequate quality to be used?				
STATISTICAL TEST Have all outliers been evaluated to determine the possible cause?				
Does data verify the assumptions in the statistical sampling plan?				
Are planned confidence intervals met?				
DECISION DETERMINATION Was this data generated by Procedure "Quality Assured Data" and is data "Data of Known Quality?" Yes--Data of Known Quality No--Information Only Data				
Can current decision be made from this data based on the above review?				

Data Assessment Performed By: _____ Date: _____
(The Assessment has been performed and data can be made available for final reporting.)

This form shall be completed and filed in the program/project data file. **Provide copy to the Data Manager for data completion.**

Attachment J
PADUCAH BJC DATA RELEASE TO EXTERNAL AGENCIES
Page 1 of 2

A one-page form located on the following page without page headers may be printed and used directly.

PADUCAH DATA RELEASE TO EXTERNAL AGENCIES

Requester:	Program/Project Title:
Dataset:	
Description of Program/Project:	

Data Quality Level:

? Data of Known Quality—Data, along with appropriate laboratory, verification, validation, and assessment qualifiers, can be used for decision making purposes and was collected and managed per procedure PA-5003.

? Information Only Data—Data quality is not assured and may or may not contain the appropriate qualifiers; however, data can be used for informational purposes or can be used for decision making with relevant documentation.

? Approval for Release to:

1. _____
2. _____
3. _____

(List specific organizations to release to.)

Explanation (Attach necessary documentation for additional release criteria.):

Requester (or Designated Alternate): _____ Date: _____
(Data is approved for release.)

Site ADC: _____ Date: _____

Site TIO: _____ Date: _____

The original form is to be included in the data assessment package and provided to the Bechtel Jacobs Company Data Manager. If this dataset is not being released to any External Agencies, single-line through and initial this form.

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Effective Date 4/23/2006

PROCEDURE TO BE TRANSITIONED:

Paducah Site-Wide
Procedure Type
(Company, Functional, Project, Subcontractor)

PA-5004: Sample Tracking, Lab Coordination, & Sample Handling Guidance
(Procedure Number and Title)

1/5/1998
(Procedure Date)

Rev. 1
(Rev. Number)

Reason for Changes: Transitioning BJC Procedures to PRS interim Procedures

SECTION	DESCRIPTION OF CHANGE
General	When reference made the Lockheed Environmental Management and/or Bechtel Jacobs LLC personnel or contract, change the reference to Paducah Remediation Services (LLC) personnel or contract. Note that the procedure numbers will not change until after the first revision by PRS LLC.
General	Change Bechtel Jacobs LLC Environmental Management (EM) Projects to Paducah Remediation Services (LLC) Environmental Management (EM) Projects
General	Attached is a cross walk from the Lockheed Environmental Management and the old Bechtel Jacobs LLC procedure numbers to the current Bechtel Jacobs LLC procedures. The procedure will be re-numbered with it is revised after transition.

Initiator/Originator: Darryl Poteet / [signature] — 4/7/06
(Print Name / Sign Name) — Date

REVIEWED BY: [signature] — 4/7/06
Discipline/Task Manager — Date

APPROVED BY: Diane McDaniel — 4/7/06
Project Manager — Date

BJC to PRS Position/Title/Organization Matrix
for PA-5004 Sample Tracking, Lab Coordiantion, & Sample Handling Guidance

Bechtel Jacobs Company Position/Title/Organization		Paducah Remediation Services Position/Title/Organization
Data Requester	→	Project Manager
EMEF Sample Manager	→	Environmental Monitoring/Sample Manager
Field Task Manager	→	Field Lead
EMEF Data Manager	→	Data Manager/Network Specialist
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BECHTEL JACOBS

BECHTEL JACOBS COMPANY LLC

REVISION ORDER A

Effective Date April 1, 1998

PROCEDURE TO BE CHANGED:

BJC-ES-01	PMSA-1102 Sample Tracking, Lab Coordination, And Sample Handling Guidance
(Manual Number)	(Procedure Number and Title)
01/15/1998	1
(Procedure Date)	(Rev. Number)

Reason for Change: To comply with Bechtel Jacobs Company LLC requirements

SECTION	DESCRIPTION OF CHANGE
Entire Document	• Replace "Lockheed Martin Energy Systems, Inc. (LMES)" with "Bechtel Jacobs Company LLC."
Acronyms	• Delete "LMES Lockheed Martin Energy Systems." Add: "Bechtel Jacobs Company LLC."
Entire Document	• Replace LMES Position Titles with Corresponding Bechtel Jacobs Company LLC Position Titles
	•
Appendix B	Appendix B. Preservation requirements must be corrected for orthophosphate when this SOP is revised after a full technical review. This parameter cannot be taken for analysis from a preserved container,

INITIATED BY: Rosemary J. Holdsworth — 03/02/1998
(Type or Print Name) — Date

REVIEWED BY: (OPTIONAL) Mike Ryan
Review/Concurrence, if needed by other department — Date
(Sign and Type or Print Name)

APPROVED BY: R.B.Barber
Functional Manager — Date
(Sign and Type or Print Name)

LOCKHEED MARTIN ENERGY SYSTEMS, INC.

Paducah Environmental Management and Enrichment Facilities
Management Control (Administrative) Procedure

SAMPLE TRACKING, LAB COORDINATION, AND SAMPLE HANDLING GUIDANCE

PMSA-1102
Revision 1
(Supersedes FOPs CP4-ER-Q1002 and CP4-ER-SAM2004)
(Supersedes PTER-1001, PTER-2032, PTER-2060, and PTSA-2004)

Prepared by: T. L. Brindley/A. M. Bosch

Approved by: _____/
P. A. Gourieux Date
Program Planning and Integration Manager

Effective Date: 01/15/98

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FOR IN-HAND USE: (Check One)	PROCEDURE CLASS: (Check One)
Yes__ No_x	__ Safety-Related
If "Yes," check one: Total__ Partial	_x_ Quality-Related
(See APPLIES TO section for details)	__ Nonquality/Nonsafety-Related

VALIDATION: Date 01/08/98 Method: Performance__ Walkthrough/Simulation__ Talkthrough_x

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PURPOSE

1.1 This procedure describes the process for coordinating sample collection and analysis, tracking sample shipment and analysis, maintaining a repository of sample and data records, and use of the Paducah Project Environmental Measurements System (PEMS).

1.2 This procedure also provides guidance on sample handling requirements related to sample container selection, sample preservation, and maximum holding time prior to analysis.

APPLIES TO

2.1 This procedure applies to all sampling and analysis activities coordinated by the Paducah Environmental Management and Enrichment Facility (EMEF) organization for the United States Department of Energy (DOE) at and near the DOE-owned Paducah Site.

OTHER DOCUMENTS NEEDED

3.1 Paducah EMEF procedure PMSA-1001, *Quality Assured Data*

3.2 Paducah EMEF procedure PTSA-2002, *Sample Chain of Custody*

3.3 Paducah EMEF procedure PTSA-2007, *Preparation of Field Quality Control Samples*

WHAT TO DO

4.1 **Planning and Preparation Activities**

Data Requester

NOTE: The relationship between the Data Requester and the Paducah EMEF Sample Manager begins upon the determination of a need for analytical services.

4.1.1 Determine various aspects associated with a data collection project independent of this procedure.

4.1.2 Provide the Paducah EMEF Sample Manager with requests or plans that describe the analytical services required.

4.1.3 **IF** a project (such as Mixed Waste Characterization, TCLP, and Vortec) generates a Sampling and Analysis plan, **THEN**

include a concurrence signature line on the plan for the Paducah EMEF Sample Manager.

Data Requester	**4.1.4**	**IF** a project (such as PCB Waste Characterization, Storage Operations, and Site Characterizations) generates a sampling request form, **THEN** provide a copy of the request form to the Sample Manager.
Paducah EMEF Sample Manager or Designee	**4.1.5**	Log the project into the Paducah EMEF Analytical Project Tracking System.
	4.2	**Coordination of Analytical Services**
Paducah EMEF Sample Manager	**4.2.1**	Review the information provided <u>and</u> work with the Data Requester to determine the analytical provider from the following: • Analytical Services Organization (ASO) laboratory • Commercial laboratory • Lockheed Martin Utility Services, Inc., (LMUS) laboratory • Paducah EMEF field laboratory
	4.2.2	**IF** the EMEF Field Laboratory is selected, **THEN** prepare an STD for the field laboratory **AND** proceed to Section 4.3.
	4.2.3	**IF** an ASO, commerical, or LMUS analytical laboratory is selected, **THEN** perform the following actions: **4.2.3.1** Enter the required information into the Oak Ridge Sample Management Organization (OR-SMO) TRACKER system. **4.2.3.2 IF** data collection activities are routine for a program, **THEN** enter an <u>annual</u> Statement of Work (SOW) into the OR-SMO TRACKER system. (Individual

SOWs are not required.)

4.2.3.3 IF data collection activities are detailed, **THEN**
prepare an individual SOW to ensure that the laboratory can meet all requirements

4.2.4 Perform the Buy/Make Analysis in TRACKER.

Paducah EMEF Sample Manager and Project Team Representatives

4.2.5 Support the laboratory Readiness Review for the project, if required.

4.2.6 Provide additional information requested by the laboratory, such as Sampling and Analysis Plans, Quality Assurance (QA) Project Plans, and Waste Management Plans.

Paducah EMEF Sample Manager

4.2.7 **IF** an LMUS laboratory is chosen, **THEN** perform the following steps:

4.2.7.1 determine whether the scope of work is covered by the current Memorandum of Understanding (MOU).

4.2.7.2 Provide either a SOW for laboratory signature **OR** notify the laboratory about the project and approve the project for laboratory acceptance.

4.2.7.3 Obtain commitment from the LMUS laboratory to perform the work.

4.2.8 **IF** an ASO lab is chosen, **THEN** contact the ASO Paducah representative to provide the SOW and any other necessary information, such as Sampling and Analysis Plans, QA Project Plans, and Waste Management Plans.

4.3 Coordination of Sample Collection and Shipment

Field Task Manager

4.3.1 Develop sampling schedule and train staff on proper procedures for sample identification, custody, and labeling.

NOTE: Not all projects will require the use of PEMS at the field level; however, all data will reside in PEMS during the data review cycle.

4.3.2 Contact the Paducah EMEF Sample Manager to establish a PEMS system for use during sample collection.

4.3.3

4.3.4 Specify the Quality Control (QC) samples (such as trip blanks, field blanks, equipment rinsates, duplicate, and matrix spike/matrix spike duplicates) that are identified by planning using Paducah EMEF procedure PMSA-1001, *Quality Assured Data*, and are collected according to Paducah EMEF procedure PTSA-2007, *Collection of Field Quality Control Samples.*

4.3.5 Ensure that Paducah EMEF procedure PMSA-1001, *Quality Assured Data*, is followed throughout the sample collection process to ensure data quality.

4.3.6 Ensure that chain-of-custody (COC) forms for samples are properly completed according to Paducah EMEF procedure PTSA-2002, *Sample Chain of Custody*.

4.3.7 Coordinate shipment or delivery of samples to the appropriate laboratory. (This includes obtaining proper radioactive screening tests and Department of Transportation [DOT] approvals.)

4.4 Tracking Sample Shipment and Analysis

Field Task Manager

4.4.1 Utilize PEMS or other approved system for sample tracking and data management activities.

4.4.2 Provide a copy of the COC to the Paducah EMEF Sample Manager upon shipment or delivery to the laboratory.

Paducah EMEF Sample Manager

4.4.3 **IF** a commercial laboratory is being used., **THEN** provide a copy of the COC to the Oak Ridge Sample Management Office.

Field Task Manager

4.4.4 Ensure that laboratory received the samples.

	4.4.5	Log the date that the sample was received by the laboratory into PEMS or other established sample tracking system.
Paducah EMEF Sample Manager	**4.4.6**	Track the receipt of data packages <u>and</u> organize for submittal to the record storage center.
	4.4.7	Coordinate data validation and laboratory resubmissions by the laboratory to the data validator.
4.4.8	**4.4.9**	Provide electronic data deliverables to the Paducah EMEF Data Manager.
	4.4.10	Perform contractual screening on all deliverables.

4.5 Maintain Logbooks and Sampling Records

Field Task Manager <u>and</u> Paducah EMEF Sample Manager	**4.5.1**	Maintain a logbook of sample shipments, other sample information, and communications with the laboratories.
	4.5.2	Maintain records certifying the quality of deionized water and potable water.
	4.5.3	Coordinate maintenance of COC records <u>and</u> sort, as needed, to provide a ready reference to completed sampling events.
	4.5.4	**IF** PEMS is being utilized for the project, **THEN** ensure that the data system is maintained accurately **AND** ensure that all logbooks are completed.
	4.5.5	Maintain all field data (if generated) accurately.

RECORDS

5.1 General Requirements

Records generated as a result of this procedure shall be processed and maintained in accordance with requirements in Paducah EMEF procedure PMIO-1000, *Paducah LMES Records Management Program*.

5.2 **Quality Records**

5.2.1 Analytical statements of work

5.2.2 Chain-of-custody forms

5.2.3 Logbooks

5.2.4 Water quality records

5.3 **Nonquality Records**

5.3.1 PEMS Data Reports

SOURCE DOCUMENTS

6.1 Paducah EMEF procedure PMIO-1000, *Paducah LMES Records Management Program*

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Definitions/Acronyms

1.0 Definitions

1.1 Contractual Screening - A process of evaluating a set of data against the requirements specified in the SOW to ensure that all requested information is received. The contractual screening includes, but is not limited to, the chain of custody (COC), number of samples, analytes requested, total number of analyses, method used, quality control samples analyzed, electronic data deliverables, units, holding times, and reporting limits achieved.

1.2 Data Requester - A project manager (or his designated representative, such as a technical lead, risk assessor, waste management coordinator, compliance coordinator, or other individual who determines the need for data to support decision making. The requester is responsible for coordinating sample collection, sample analysis, data assessment, and decision making. If the requestor is a designated representative, the project manager has ultimate responsibility.

1.3 Field Task Manager - The Field Task Manager is responsible for ensuring the collection and delivery to the appropriate laboratory of all required environmental samples. The Field Task Manager is also responsible for ensuring that field records (field logbooks, forms, and COC records) are maintained.

1.4 Paducah EMEF Analytical Project Tracking System - A Paducah EMEF system developed to track sampling requests and project by assigning either a system generated project number for some programs or allowing the user to assign a unique project number. The system is maintained by the Paducah EMEF Sample Manager to track various information about the event such as the charge number, sampling time frame, status, etc.

1.5 Paducah EMEF Data Manager - The Data Manager is responsible for long term electronic storage of data, for loading electronic data deliverables, electronic verification of data, and ensuring compliance to the EMEF Data Management Policy.

1.6 Paducah EMEF Sample Manager - The Sample Manager is responsible for interfacing with the Oak Ridge Sample Management Office for procurement of laboratory services and/or validation services and coordinating contractual screening.

1.7 Paducah PEMS - The Paducah data base system used for field preparation and tracking of data collection activities. This system is provided to project personnel and pre-populated with project information. The system generates COCs, bottle labels, and other field forms; stores analysis type and preservative information; tracks sampling progress; and stores location information.

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1.8 **TRACKER** - This data base system is the business operation system of the OR-SMO. The system generates SOWs, documents the buy/make process, tracks sample shipments, tracks billing, data deliverables, and sample disposal.

2.0 Acronyms

2.1 **ASO** - Analytical Services Organization

2.2 **COC** - Chain of Custody

2.3 **DOE** - Department of Energy

2.4 **DOT** - Department of Transportion

2.5 **EMEF** - Environmental Management and Enrichment Facilities

2.6 **LMUS** - Lockheed Martin Utility Services, Inc.

2.7 **MOU** - Memorandum of Understanding

2.8 **OR-SMO** - Oak Ridge Sample Management Office

2.9 **PCB** - Polychlorinated Biphenyl

2.10 **PEMS** -- Project Environmental Measurements System

2.11 **QA** - Quality Assurance

2.12 **QC** - Quality Control

2.13 **SOW** - Statement of Work

2.14 **TCLP** - Toxicity Characterization Leaching Procedure

Appendix B
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Recommended Holding Times, Containers, and Preservation for Analytical Samples

Parameter	Method No.	Matrix	Holding Time (from time collected)	Sample Container*	Preservation
Volatile organics	SW-846[1], 8240/8260 or 8081 Prep 5030	Water	14 days	Three 40-mL glass vials with Teflon-lined septum	HCl to pH<2[2] Cool to 4°C
		Soil	14 days	4-oz. widemouth glass jar with Teflon-lined closure or brass liner	Cool to 4°C
Aromatic volatile organics	SW-846, 8020 Prep 5030	Water	14 days	Three 40-mL amber glass vials with Teflon-lined septum	HCl to pH<2 Cool to 4°C
		Soil	14 days	4-oz. widemouth glass jar with Teflon-lined closure or brass liner	Cool to 4°C
Methyl Ethyl Ketone	SW-846, Modified 8015, Prep 5030	Water	14 days	Three 40-Ml amber glass vials with Teflon-lined septum	HCL to pH<2 Cool to 4°C
		Soil	14 days	8-oz. widemouth jar with Teflon-lined closure	Cool to 4°C
Semivolatile organics	SW-846, 8270 Water Prep. 3510/3520	Water	7 days to extraction 40 days to analysis	Two 1-L amber glass jars with Teflon-lined closure	Cool to 4°C
	Soil Prep. 3540/3550	Soil	14 days to extraction 40 days to analysis	8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
	Biota Prep[4]	Fish, grass	14 days to extraction 40 days to analysis	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Freeze w/dry ice
Metals[5] (other than mercury)	SW-846, 6010 Arsenic-7060, Lead-7421, Selenium-7740, Thallium-7841, Water Prep 3010	Water	180 days	1-L polyethylene with polyethylene closure	HNO_3 to pH<2 Cool to 4°C
	Soil Prep 3050	Soil	180 days	8-oz. widemouth glass jar with Teflon-lined closure	Freeze w/dry ice
	Biota Prep[4]	Fish, grass	180 days	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	

NOTE: See page 6 of this appendix for all footnotes

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Parameter	Method No.	Matrix	Holding Time (from time collected)	Sample Container*	Preservation
Mercury[a]	SW-846, 7470	Water	28 days	2-L polyethylene with polyethylene closure	HNO_3 to pH<2
	SW-846, 7471	Soil	28 days	8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
	Biota Prep[c]	Fish, grass	28 days	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Freeze w/dry ice
Total Lead	SW-846, 7421 Water Prep 3020	Water	6 months	1-L polyethylene with polyethylene closure	HNO_3 to pH<2
	Soil Prep 3050	Soil	6 months	8-oz. glass jar with Teflon-lined closure	None
	Biota Prep[c]	Fish, grass	6 months	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Freeze w/dry ice
Cyanide	SW-846, 9010	Water	14 days	1-L polyethylene or glass with Teflon-lined closure	NaOH to pH>12
		Soil	14 days	4-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
	Biota Prep[c]	Fish, grass	14 days	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Cool to 4°C Freeze w/dry ice
Pesticides/ PCBs	SW-846, 8080 Water Prep 3510/3520	Water	7 days to extraction 40 days to analysis	Two 1-L glass jars with Teflon-lined closure	Cool to 4°C
	Soil Prep 3540/3550	Soil	14 days to extraction 40 days to analysis	16-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
	Biota Prep[c]	Fish, grass	14 days to extraction 40 days to analysis	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Freeze w/dry ice
Dioxins/Furans	SW-846, 8280	Water	30 days to extraction 45 days to analysis	1-L amber glass with Teflon-lined closure	Cool to 4°C
		Soil	30 days to extraction 45 days to analysis	4-oz. widemouth amber glass jar with Teflon-lined closure or brass liner	Cool to 4°C
	Biota Prep[c]	Fish, grass	30 days to extraction 45 days to analysis	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Freeze w/dry ice

NOTE: See page 6 of this appendix for all footnotes

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Parameter	Method No.	Matrix	Holding Time (from time collected)	Container	Preservation
Gross Alpha and Beta[a]	SW-846, 9310	Water	6 months	Two l-gal polyethylene with polyethylene closure	HNO_3 to pH<2
		Soil	6 months	8-oz. widemouth amber glass jar with Teflon-lined closure	Cool to 4°C
	Biota Prep[c]	Fish, grass	6 months	Wrap no less than 30 grams of specimen in aluminum foil and seal in ziplock baggie	Freeze w/dry ice
Other Radionuclides U-234 U-238 Tc-99 Np-237 Pu-239	 EERF 00-07[e] EERF 00-07 MMES R-46[f] HASL 300-E-NP-01[g] EERF 00-07	Water Soil	6 months 6 months	Two l-gal polyethylene with polyethylene closure 8-oz. widemouth glass jar with Teflon-lined closure	HNO_3 to pH<2 None
Acute Toxicity Test[h]	EPA/600/4-85/013[i]	Water	36 hours	2-L polyethylene with polyethylene closure	Cool to 4°C
Chronic Toxicity Test[h]	EPA/600/4-89/001[j]	Water	36 hours	2-L polyethylene with polyethylene closure	Cool to 4°C
Ammonia-nitrogen[k] Orthophosphate Nitrogen (Total) Total Organic Carbon	ASTM[l] ASTM D 4183 ASTM E 778 SW-846[a], 9060	Soil	28 days 48 hours[m] 28 days 28 days	16-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C Cool to 4°C Cool to 4°C Cool to 4°C
Cr VI	SW-846, 7195 SW-846, 7196	Soil Water	24 hours[n] 24 hours[n]	8-oz. widemouth glass jar with Teflon-lined closure 250-mL polyethylene with polyethylene closure	Cool to 4°C Cool to 4°C
Sulfate[o] Sulfide	SW-846, 9035 SW-846, 9030	Soil	28 days 28 days	16-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C Cool to 4°C
Particle-size Distribution[p], Moisture Content, pH	ASTM D422 ASTM D2216 SW-846, 9045	Soil	None 14 days 48 hours	Two 16-oz. widemouth glass jars	None Cool to 4°C Cool to 4°C
Flash Point	SW-846, 1010	Soil	None specified	8-oz. widemouth glass jar with Teflon-lined closure	None
Ammonia[q] Nitrate - Nitrite Total Kjeldahl Nitrogen Orthophosphate Phosphorous (Total)	MCAWW[r]-350.2 SW-846, 9200 MCAWW-351.3 MCAWW-365.2 MCAWW 365.2	Water Water Water Water Water	28 days 28 days 28 days 48 hours[m] 28 days	2-L polyethylene with polyethylene closure	H_2SO_4 to pH<2 Cool to 4°C
Hardness (Total)	MCAWW-130.1	Water	28 days	250-mL polyethylene with polyethylene closure	HNO_3 to pH<2 Cool to 4°C

NOTE: Refer to page 6 of this appendix for all footnotes.

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Parameter	Method No.	Matrix	Holding Time	Container	Preservation
Total Organic Carbon Chemical Oxygen Demand	SW-846, 9060 MCAWW-410.1	Water Water	28 days 28 days	2-L polyethylene with polyethylene closure	H_2SO_4 to pH<2 Cool to 4°C
Sulfide	SW-846, 9030	Water	7 days	2-L polyethylene with polyethylene closure	ZnAc & NaOH to pH>9 Cool to 4°C
Total Suspended Solids[a] Sulfate Fluoride pH Ignitability Chloride	MCAWW-160.2 SW-846, 9035-9038 MCAWW-340.2 SW-846, 9045 SW-846, 1010 MCAWW 325.3	Water Water Water Water Water Water	7 days 28 days 28 days At collection None specified 28 days	2-L polyethylene with polyethylene closure	Cool to 4°C Cool to 4°C None None None None
Biochemical Oxygen Demand	MCAWW-405.1	Water	48 hours	1-L polyethylene with polyethylene closure	Cool to 4°C
Oxidation/Reduction Potential (Eh)	ASTM D 1498	Water	48 hours	16-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
BTU Content[c]	ASTM[d] D246	Soil	14 days	8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
Ash Content[c]	ASTM E830	Soil	14 days	8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
Moisture Content[c]	ASTM D2216	Soil	14 days	8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
Sulfur[e] (Total)	ASTM 4468 ASTM D4239	Water Soil	28 days 28 days	1-L polyethylene with polyethylene closure 8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C Cool to 4°C
Chloride[e]	MCAWW 325.3 SW-846, 9252	Water Soil	28 days 28 days	1-L polyethylene with polyethylene closure 8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C Cool to 4°C
Fluoride[e]	MCAWW 340.2	Water Soil	28 days 28 days	1-L polyethylene with polyethylene closure 4-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C Cool to 4°C
Flash Point	SW-846, 1010	Soil	None Specified	8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
Ignitability	SW-846, 1010	Water	None Specified	125-mL polyethylene with polyethylene closure	Cool to 4°C
Paint Filter Test	SW-846, 9095	Soil	48 hours	16-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C
Particle Size Distribution	ASTM D422	Soil	None	16-oz. widemouth glass jar with Teflon-lined closure	None

NOTE: See page 6 of this appendix for all footnotes.

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Parameter	Method No.	Matrix	Holding Time	Container	Preservation
Full TCLP	SW-846, 1311 followed by SW-846, 8240/8260, 8720, 8080, 8150, 6010, 7470	Soil	VOCs: 14 days to extraction SVOCs and pesticide/PCBs: 14 days to extraction Mercury: 28 days to extraction Metals: 180 days to extraction	16-oz. widemouth glass jar with Teflon-lined closure*	Cool to 4°C
Reactivity	Chapter 7 of SW-846	Water Soil	None Specified	1-L polyethylene with polyethylene closure 8-oz. widemouth glass jar with Teflon-lined closure	Cool to 4°C Cool to 4°C

NOTE: See page 6 of this appendix for all footnotes.

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Footnotes:

- * Additional volume may be needed for matrix spike/matrix spike duplicate samples. For aqueous samples: double volume will be collected for inorganics; triple volume will be collected for organics. No extra volume is required for soil/sediment samples.
- [a] U.S. Environmental Protection Agency, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, November 1986b.
- [b] The first sample for volatile analysis will be tested for pH by first adding four drops of HCl to a spare 40-mL vial, adding the water sample, and testing with pH paper. If pH>2, additional acid will be added until pH<2 is achieved. Once this determination has been made, remaining VOA vials will be pre-preserved before sample collection.
- [c] Biota (fish tissue and grass) preparation for analysis will be according to laboratory standard operating procedures due to the variability in biota preparation methods.
- [d] Samples for these parameters can occupy the same sample containers.
- [e] U.S. Environmental Protection Agency, *Eastern Environmental Radiation Facility, Radiochemistry Procedures Manual*, 520/5-84-006, 1984.
- [f] Martin Marietta Energy Systems, *Tc-99 Determination in Environmental Water and Low Level Contaminated Water (MEK Extraction)*, R-46 Method, June 15, 1990b.
- [g] Department of Energy, *EML Procedures Manual, Environmental Measurements Laboratory*, 25th Edition, 1982.
- [h] Tests will be conducted on *Ceriodaphnia dubia and Pimephales promelas* (fathead minnows).
- [i] U.S. Environmental Protection Agency, *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater Organisms*, EPA/600/4-85-013, 1985.
- [j] U.S. Environmental Protection Agency, *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms*, EPA/600/4-89/001, 1989h.
- [k] Samples for these parameters can occupy the same sample containers.
- [l] American Society for Testing and Materials, *Annual Book of ASTM Standards*, 1991 Ed. Method will be determined by analytical laboratory.
- [m] The laboratory will be required to meet 48-hour holding time; laboratory will report potential problems concerning holding time compliance immediately so that corrective action can be taken.
- [n] Twenty-four hours is the goal for holding time. If needed, the laboratory will be permitted 24 hours; analytical results exceeding the 24-hour holding time will be flagged as estimated.
- [o] Samples for these parameters can occupy the same sample container.
- [p] Samples for these parameters can occupy the same sample container.
- [q] Samples for these parameters can occupy the same sample container.
- [r] U.S. Environmental Protection Agency, *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, March 1983b.
- [s] Samples for these parameters can occupy the same sample container.
- [t] Samples for these parameters can occupy the same sample container(s).
- [u] Samples for these parameters can occupy the same sample container(s).
- [v] Sample volume for TCLP analysis varies with the percent solid. If percent solid is <50%, up to 5 L may be required. Consult with the laboratory completing the TCLP analyses prior to collection of TCLP samples to verify volume requirement.

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PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

OWNER: Tim Stout	**PRS-WSD-9503**	**REV. NO. 0**
SUBJECT MATTER AREA: Sample Shipments	**PREPARER: David DeLaCruz**	**Page 1 of 19**
DOC TYPE: ☒ **PROCEDURE** ☐ **POLICY**	**APPROVED BY/DATE:** **Tim Stout (Signature of File in DCC)** **3/14/07**	
PROC TYPE: ☒ **OPERATING PROCEDURE** ☐ **FACILITY SPECIFIC PROCEDURE** **FACILITY: _____**		
TITLE: **OFF-SITE SAMPLE SHIPPING**		
USQD ☒ **UCD** ☒ **CAT EX** ☐	**EFFECTIVE DATE: 09/20/06**	
USQD/UCD No: USQD-PH-WASTE-0042 / UCD-PH-WASTE-0082	**REQUIRED REVIEW DATE: 09/20/09**	
Mandatory Subcontractor Pro Forma Procedure? ☐	**If an interim Procedure, Expiration Date:**	

REVISION LOG		
Revision Number	Description of Changes	Pages Affected
0	Initial Release. Intent Change. Changed numbers and headings to define the beginning point of Paducah Remediation Services documentation and to establish Document Control as the control point for tracking document numbers. This document replaces PRS blue-sheeted TR-9503.	All

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1.0 PURPOSE

This procedure provides instructions for preparing shipments of hazardous and non-hazardous material samples to ensure that they are properly shipped. This procedure provides the processes to satisfy the requirements related to sample shipment in the following:

- 49 Code of Federal Regulations (CFR), *Transportation* (Parts 100-177)
- International Air Transportation Association (IATA)

2.0 SCOPE

The requirements of this procedure apply to all off-site sample shipments performed by Paducah Remediation Services, LLC (PRS) on behalf of the United States Department of Energy (DOE) at the DOE-owned Paducah Site.

NOTE 1: ALL regulated sample shipments containing hazardous materials must be prepared and/or reviewed by an IATA trained individual.

3.0 PROCEDURE

3.1 Materials

Certified Shipper (or designee)

Obtain the special tools, equipment and sample shipment supplies needed including the following, as applicable:

- Shipping containers
  a. cooler packs
  b. excepted packaging
  c. 1 A2/X or Y drums
- Artificial Ice
- Absorbent packing material
- Bubble wrap
- Plastic bags (i.e. quart size)
- Tamper indicating tape
- Reinforced tape
- Assortment of labels (i.e., Corrosive, Package Orientation, and Cargo Aircraft Only.)

NOTE 1: Characterization samples generally require temperature controls and/or preservation media.

3.2 Pre-Shipment Activities

Certified Shipper (or designee)

3.2.1 Obtain a Radiological Shipment Survey on the shipping container(s) and sample bottles/containers.

3.2.2 Choose a carrier and ensure State and Carrier Limitations (per IATA, Section 2, Dangerous Goods Regulations) are met.

3.3 Laboratory

Certified Shipper (or designee)

3.3.1 Ensure that the laboratory is approved by the PRS Sample Management Organization (SMO).

3.3.2 If the samples being shipped contain radiological material, notify the lab(s) prior to shipment using the *Acceptance of Sample Materials* form (Form #: WSD-F-0059).

NOTE 1: The *Acceptance of Sample Materials* form is not required for materials that are considered non-radiological (no rad added or free-releasable). In this case, an e-mail or equivalent form of confirmation from the lab is acceptable.

NOTE 2: Samples being sent to non-NRC licensed facilities must be free-released by the Radiation Technician.

3.3.3 **PRIOR** to shipment, ensure that confirmation has been received, either by telephone, fax, or e-mail that the lab can accept the samples.

3.4 Radiological Characterization

3.4.1 Determine if the material has no added rad (per process knowledge (PK); or if the material is essentially equivalent to background).

3.4.2 Determine if the material is surface contaminated.

NOTE 1: Contamination means the presence of a radioactive substance on a surface in quantities in excess of 0.4 Bq/cm^2 (2,400 dpm/100 cm^2) for beta and gamma emitters and low toxicity alpha emitters; or 0.04 Bq/cm^2 (240 dpm/100 cm^2) for all other alpha emitters.

NOTE 2: If contamination is present above the levels specified above, the material must be regulated as Class 7. The PRS Transportation Group shall be contacted for assistance in determining the Proper Shipping Name (PSN) and shipping requirements.

NOTE 3: Material with Fixed Beta/Gamma contamination less than 4,000 dpm/100 cm^2 may be free-released, but the limit for contamination, as described by DOT, is 2,400 dpm/100 cm^2; which means that material that is free-released may still be regulated as Class 7.

3.4.3 Determine if the material is volumetrically contaminated per results from a rad survey, PK, or historical data. Calculations must be performed in accordance with 49 CFR 173.403/IATA 10.3.1 to determine if the material is:

- Non-regulated as Class 7 by DOT/IATA

- Regulated as Class 7 and can be shipped as limited quantity, or
- Regulated as Class 7 and can be shipped as low specific activity (LSA) or surface contaminated objects (SCO)

NOTE 1: If calculations confirm that the material should be regulated as Class 7, the PRS Transportation Group shall be contacted for assistance in determining the PSN and shipping requirements.

NOTE 2: Radionuclides will be listed on shipping papers utilizing PK for radioactive material shipments when only gross alpha/beta data has been provided.

NOTE 3: For all regulated sample shipments of radioactive material (Class 7 as primary or subsidiary hazard), calculations and associated shipping documents must be peer reviewed by a qualified shipper from the PRS Transportation Group prior to release.

3.5 Additional General Characterization Requirements

Certified Shipper (or designee)

3.5.1 All software, spreadsheets and/or databases must be quality checked for accuracy. Testing and validation of computer programs and verification of data results from their programs shall be conducted and documented in accordance with BJC-IT-6007, *Software Quality Assurance Program*.

3.5.2 Complete a Sample Shipment Preparation Checklist (Form #: WSD-F-0060), and include in the shipment file.

NOTE 1: This checklist should be completed for all shipments, hazardous and non-hazardous.

3.5.3 Ensure a full peer review, including calculations, is completed for each regulated sample shipment of radioactive material.

NOTE 1: Guidance to determine the appropriate documentation to be retained in the Shipment File, for typical sample shipment types, is provided in Attachment B.

3.5.4 Use the *Limited Quantity Radioactive Materials Checklist/Peer Review* form (Form #: WSD-F-0061) when a material is being shipped as a Limited Quantity in an Excepted Package.

3.5.5 Use one of the current checklists as provided in the IATA regulations as appropriate. Examples are provided as follows:

- *Dangerous Goods Checklist for a Non Radioactive Shipment* (Attachment C)
- *Dangerous Goods Checklist for a Radioactive Shipment* (Attachment D)

3.5.6 Initiate a shipment utilizing the following information in one or more of these formats:

- Process Knowledge (PK) and Health Physics (HP) surveys
- Data and HP Surveys
- Radiological analysis and HP Surveys

3.5.7 Ensure the following information is available:

- Sample Identification number(s)
- Matrix
- Container Type (e.g. Glass 40 ml bottle)
- Quantity (net of material)
- Preservation Media, if any
- Delivery schedule
- Known possible hazardous constituents (Radiological activity, acids as preservatives, etc.)
- Number of blanks to be shipped with the samples
- Physical Form (i.e. liquid, solid, gas)
- Chemical Form (i.e. oxides or nitrates)
- Technical Name
- Industrial Hygiene (IH) Samples

3.5.8 Characterize the material as Dangerous Goods (hazardous), or Not Restricted (non-hazardous), as applicable.

3.6 Packaging

NOTE 1: Refer to IATA Dangerous Goods Regulations for specific packaging requirements when shipping hazardous materials by air and Department of Transportation (DOT) 49 CFR parts 100 to 185 when shipping hazardous materials by ground.

Certified Shipper (or designee)

3.6.1 Package samples by performing the following:

- Secure any friction-type closures with tape (i.e. stoppers, corks, etc.)
- Ensure liquids are surrounded by absorbent material sufficient enough to meet IATA Table 5.0 B.
- Ensure all packages with liquid in the inner packagings have package orientation labels (up arrows) on **two** opposing sides, and
- Ensure all packaging instructions are followed, secondary containment may be required.

3.6.2 Ensure the original chain-of-custody for each sample is included in the package.

3.6.3 Apply all applicable IATA and International Civil Air Organization (ICAO) labeling and markings, airway bill and Shipper's Declaration to the shipping container.

3.6.4 Ensure HP has issued an off site release survey **PRIOR** to shipping.

NOTE 1: Limited Quantity Radioactive Material shipments must include the United Nations (UN) identification number on the package.

NOTE 2: Ensure the label of each sample matches the chain-of-custody along with the list of samples to be shipped.

3.6.5 Limited Quantity Radioactive Material packages must be designed to meet the requirements of IATA Section 10.5.9.

NOTE 1: Ullage – Sufficient outage must be left to ensure that neither leakage nor permanent distortion of packaging will occur during transit. Liquids must not completely fill a packaging at 55C (130F).

NOTE 2: When samples requiring no "head space" (e.g. volatile and semi-volatile samples) are shipped, an intermediate containment MUST be used.

NOTE 3: If the inner packagings cannot meet the pressure requirements, an intermediate containment that does meet the requirements can be used.

3.7 Notification

Certified Shipper (or designee)

3.7.1 Contact carrier to arrange shipment pickup.

3.7.2 Inform carrier if material needs special handling (i.e. to be palletized).

3.8 Documentation

Certified Shipper (or designee)

3.8.1 Attach the airway bill to the package.

NOTE 1: Each air carrier has its own airway bill. For example, the airway bill for FedEx and UPS can be "labels-created" online. Emery has a hardcopy, one-half sheet bill of lading.

3.8.2 Attach the Shipper's Declaration of Dangerous Goods shipping document to the package, when applicable.

3.8.3 Send a copy of the Shipper's Declaration of Dangerous Goods airway bill to the appropriate air carrier for review prior to shipment, when required to do so by the carrier.

NOTE 1: The air carrier, Emery, specifically requests to review a Shipper's Declaration of Dangerous Goods for samples that contain hazardous material. It also requests that the airway bill is marked "NOT RESTRICTED" if the shipment does NOT

contain hazardous material (refer to step 3.5.8).

3.8.4 Send appropriate number of originals with the shipment as determined by the carrier (usually three will suffice).

3.8.5 Place signed copies of the chain-of-custody forms for each sample in the shipment file.

NOTE 1: Drums containing hazardous materials will be palletized by EMERY. Ensure all DOT UN Specification markings from the bottom of the drum are replicated on the side of the drum for visibility.

3.9 Post-Shipment Activities

Certified Shipper (or designee)

3.9.1 Retain the original signed copy of airway bill and an original of the Shipper's Declaration.

3.9.2 Notify the Plant Shift Supervisor (PSS) and provide copy of Shipper's Declaration or Bill of Lading for all regulated (hazardous) shipments.

3.9.3 Ensure all shipping containers were received at the off-site laboratory by running a package trace or by making a phone call.

3.9.4 **IF** all containers were NOT received within the expected delivery time, **THEN** initiate a trace to locate the shipment.

3.9.5 Ensure the returned shipping containers are inspected and cleaned (if necessary) for future use.

3.9.6 **IF** a DOT or other regulatory shipping violation has occurred, **THEN** initiate an occurrence report in accordance with BJC-PQ-1220 [New PRS-QAP-1220].

4.0 RECORDS

The following records may be generated by this procedure and are to be maintained in accordance with BJC-OS-1001, *Records Management, Including Document Control* [New PRS-DOC-1009, *Records Management, Administrative Records, and Document Control*]:

- Airway bill (or copy of UPS or FedEx label),
- Shipper's Declaration of Dangerous Goods, when applicable,
- Radiological data,
- Transportation calculations generated,
- Hazardous Materials checklist and peer review, or equivalent,
- HP survey of outer box, when prepared in an RMA,
- HP survey of Samples, and
- Other pertinent data (i.e. Chain of Custody copies, etc.)

5.0 SOURCE DOCUMENTS

NOTE: The PRS blue-sheeted BJC procedures referenced in this document are the active procedures as of the date of issuance of this procedure. Procedures noted in the parenthesis [brackets] will become the reference procedures once these procedures are approved and implemented by Paducah Remediation Services, LLC.

- 49 CFR, *Transportation*
- IATA Dangerous Goods Regulations, 47th Edition
- BJC-PQ-1220*, Occurrence Notification and Reporting* [PRS-QAP-1220]

Attachment A
DEFINITIONS/ACRONYMS
Page 1 of 1

DEFINITIONS

Refer to BJC/PAD-11, *Waste Acceptance Criteria for the DOE Treatment, Storage, and Disposal Units at the Paducah Gaseous Diffusion Plant, Paducah, KY* [New PRS-WSD-0011, *Waste Acceptance Criteria for the Treatment, Storage, and Disposal Facilities at the Paducah DOE Site]*.

ACRONYMS

CFR:	Code of Federal Regulations
DOE:	Department of Energy
DOT:	Department of Transportation
HP:	Health Physics
IATA:	International Air Transportation Association
LSA:	Low-Specific Activity
NRC:	Nuclear Regulatory Commission
PK:	Process Knowledge
PRS:	Paducah Remediation Services, LLC
PSN:	Proper Shipping Name
SCO:	Surface Contaminated Object
SMO:	Sample Management Organization
UN:	United Nations
UPS:	United Postal Service

Attachment B
Documentation Requirements for Typical Sample Shipments by Air
Page 1 of 2

Shipment Classification	Radiological Status	Proper Shipping Name (PSN) Examples	Required Shipping Documents	Shipping File to Include
Non-Regulated by DOT/IATA	No Rad Added[1] Free Releasable[2]	N/A	Air Waybill	● Sample Shipment Preparation Checklist ● Completed Air Waybill ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody
	Contains Rad Material[3]	N/A	Air Waybill	● Sample Shipment Preparation Checklist ● Lab Acceptance of Samples Form ● Analytical Data (Rad Screen) ● Calculations with Peer Review Signature ● Completed Air Waybill ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody
Regulated as Class 7 Radioactive	Meets Limited Quantity Requirements for Class 7	UN2910, Radioactive material, excepted package, limited quantity of material, 7	Air Waybill – Must include PSN on the waybill in the Nature and Quantity of Goods	● Sample Shipment Preparation Checklist ● Lab Acceptance of Samples Form ● Analytical Data (Rad Screen) ● Calculations with Peer Review Signature ● Completed Air Waybill ● Completed LTD QTY Checklist ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody
	Exceeds Limited Quantity Requirements, but meets appropriate LSA Requirements	UN2912, Radioactive material, low specific activity (LSA-I), 7	Air Waybill Shipper's Declaration of Dangerous Goods	● Sample Shipment Preparation Checklist ● Lab Acceptance of Samples Form ● Analytical Data (Rad Screen) ● Calculations with Peer Review Signature ● Completed Air Waybill ● Completed Dangerous Goods Declaration ● Completed IATA Radioactive Checklist ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody

Attachment B
Documentation Requirements for Typical Sample Shipments by Air
Page 2 of 2

Not Class 7 but Regulated as Another DOT Class (Ex. Corrosives – Class 8, Miscellaneous – Class 9, etc.)	No Rad Added or Free Released (not Class 7)	UN3082, Environmentally hazardous substance, liquid, n.o.s., 9, (PCB)	Air Waybill	● Sample Shipment Preparation Checklist ● Analytical Data (if applicable for non-rad constituents) ● Completed Air Waybill ● Completed Dangerous Goods Declaration ● Completed IATA Non-Radioactive Checklist ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody
	Contains Rad Material and meets Limited Quantity Requirements for Class 7	UN1760, Corrosive Liquid n.o.s., 8 (7), (Nitric Acid Solution)	Air Waybill Shipper's Declaration of Dangerous Goods	● Sample Shipment Preparation Checklist ● Lab Acceptance of Samples Form ● Analytical Data (Rad Screen and other data as applicable) ● Calculations with Peer Review Signature ● Completed Air Waybill ● Completed Dangerous Goods Declaration ● Completed IATA Non-Radioactive Checklist ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody
	Contains Rad Material but doesn't meet Class 7 Requirements	UN3082, Environmentally hazardous substance, liquid, n.o.s., 9, (PCB)	Air Waybill Shipper's Declaration of Dangerous Goods	● Sample Shipment Preparation Checklist ● Lab Acceptance of Samples Form ● Analytical Data (Rad Screen and other data as applicable) ● Calculations with Peer Review Signature ● Completed Air Waybill ● Completed Dangerous Goods Declaration ● Completed IATA Non-Radioactive Checklist ● HP Survey for Release ● Confirmation of Sample Delivery ● Copy of signed Chains of Custody

1. No Rad Added – Either known by process knowledge (PK) never to have been in contact with radiological contamination; or essentially equivalent to background levels.
2. Free-Releasable Material – Must meet the surface criteria established in DOE Order 5400.5 (equivalent to 10CFR 835, App. D; except for Transuranic Waste, which is 100 dpm/100cm^2); or must be determined equivalent to background if the material is volumetrically contaminated. Samples being sent to non-NRC licensed facilities must be free-released by RADCON.
3. Contains Rad Material – Any radiological material that cannot be described as "No Rad Added" or "Free-releasable". Radiological material may or may not meet Class 7 requirements and be regulated by DOT. Calculations will be performed to determine this.

Attachment C
DANGEROUS GOODS CHECKLIST FOR A NON-RADIOACTIVE SHIPMENT
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DANGEROUS GOODS CHECKLIST FOR A NON-RADIOACTIVE SHIPMENT

The recommended checklist appearing on the following pages is intended to verify shipments at origin.

Never accept or refuse a shipment before all items have been checked.

Is the following information correct for each entry?

SHIPPERS DECLARATION FOR DANGEROUS GOODS (DGD)

	YES	NO*	N/A
1. Two copies in English and in the IATA format [8.1.1, 8.1.2]	❑	❑	
2. Full name and address of Shipper [8.1.6.1]	❑	❑	
3. Full name and address of Consignee [8.1.6.2]	❑	❑	
4. Name and Telephone Number of a responsible person for Division 6.2 Infectious Substance shipment [8.1.6.2]	❑	❑	❑
5. If the Air Waybill number is not shown, enter it. [8.1.6.3]	❑		
6. The number of pages shown [8.1.6.4]	❑	❑	
7. If full name of Airport or City of Departure or Destination is not shown, enter it. [8.1.6.6 and 8.1.6.7]	❑		
8. The non-applicable Aircraft Type deleted [8.1.6.5]	❑	❑	
9. The word "Radioactive" deleted [8.1.6.8]	❑	❑	
Identification			
Note: *Alternative sequence acceptable 8.1.6.9.1(b).*			
10. UN or ID Number, preceded by prefix [8.1.6.9.1(a), Step 1a]	❑	❑	
11. Proper Shipping Name and the technical name in brackets for asterisked entries [8.1.6.9.1(a), Step 2a]	❑	❑	
12. Class or Division, and for Class 1, the Compatibility Group, [8.1.6.9.1(a), Step 3a]	❑	❑	
13. Subsidiary Risk, in parentheses, immediately following Class or Division [8.1.6.9.1(a), Step 3a]	❑	❑	❑
14. Packing Group [8.1.6.9.1(a), Step 4a]	❑	❑	❑
Quantity and Type of Packing			
15. Number and Type of Packages [8.1.6.9.2, Step 5]	❑	❑	
16. Quantity and unit of measure (net or gross, as applicable) per package [8.1.6.9.2, Step 5]	❑	❑	
17. When different dangerous goods are packed in one outer packaging, the following rules are complied with:			
– Compatible according to Table 9.3.A. (note exception for chemical kits/first aid kits. See Packing Instructions 915 and Y915)	❑	❑	❑
– UN packages containing Division 6.2 [5.0.2.11(c)]	❑	❑	❑
– "All packed in one (type of packaging)" [8.1.6.9.2, Step 5(e)]	❑	❑	❑
– Calculation of "Q" value must not exceed 1 [5.0.2.11; 5.0.3.2; 8.1.6.9.2, Step 5(f)]	❑	❑	❑
18. Overpack			
– Indication "Overpack used" [8.1.6.9.2, Step 6]	❑	❑	❑
– Compatible according to Table 9.3.A. [5.0.1.5.1 and 5.0.1.5.3]	❑	❑	❑
Packing Instructions			
19. Packing Instruction Number [8.1.6.9.3, Step 7]	❑	❑	
Authorizations			
20. Indication of "Limited Quantity" or "Ltd. Qty." if "Y" packing instruction used [8.1.6.9.4, Step 8]	❑	❑	❑
21. The Special Provision Number if A1, A2, A51, A81 or A109 [8.1.6.9.4, Step 8]	❑	❑	❑
22. Indication that governmental authorization is attached, including a copy in English [8.1.6.9.4, Step 8]	❑	❑	❑
23. Additional approvals for other items under [8.1.6.9.4, Step 8]	❑	❑	❑
Additional Handling Information			
24. The mandatory statement shown for self-reactive and related substances of Division 4.1 and organic peroxides of Division 5.2, or samples thereof [8.1.6.11.1 and 8.1.6.11.2]	❑	❑	❑
25. The air transport statement included or added following Certification Statement [8.1.6.12]	❑	❑	
26. **Name and Title of Signatory, Place and Date** indicated [8.1.6.13 and 8.1.6.14]	❑	❑	
27. **Signature** of Shipper [8.1.6.15]	❑	❑	
28. **Amendment** or alteration signed by Shipper [8.1.2.6]	❑	❑	❑

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Attachment C
DANGEROUS GOODS CHECKLIST FOR A NON-RADIOACTIVE SHIPMENT
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	YES	NO*	N/A
AIR WAYBILL			
29. The Handling Information box shows: "Dangerous goods as per attached Shipper's Declaration" or "Dangerous Goods as per attached DGD" [8.2.1(a)]	❑	❑	
30. "Cargo Aircraft Only" or "CAO", if applicable [8.2.1(b)]	❑	❑	❑
31. Where non-dangerous goods are included, the number of pieces of dangerous goods shown [8.2.2]	❑	❑	❑
PACKAGE(S) AND OVERPACKS			
32. Packaging conforms with packing instruction and is free from damage or leakage [9.1.1.3]	❑	❑	
33. Same number and type of packagings and overpacks delivered as shown on DGD [9.1.1.3]	❑	❑	
Markings			
34. UN Specification Packaging, marked according to 6.0.4 and 6.0.5:			
– Symbol and Specification Code	❑	❑	❑
– X, Y or Z, agreed with Packing Group/Packing Instruction	❑	❑	❑
– Gross Weight within limits (Solids or Inner Packagings)	❑	❑	❑
– Infectious substance package marking [6.0.6]	❑	❑	❑
35. The Proper Shipping Name(s) including technical name where required, and the UN or ID Number(s) [7.1.5.1(a)]	❑	❑	
36. The full name(s) and Address(es) of Shipper and Consignee [7.1.5.1(b)]	❑	❑	
37. The Net Quantity of Explosives and Gross Weight of the package for Class 1 items [7.1.5.1(c)]	❑	❑	❑
38 Classes 2 to 6, 8 & Carbon Dioxide, Solid (Dry Ice), the net quantity or gross weight as applicable marked on the packages [7.1.5.1(d) and (e)]	❑	❑	❑
39. The Name and Telephone Number of a responsible person for Division 6.2 Infectious Substances shipment [7.1.5.1(f)]	❑	❑	❑
40. The Special Marking requirements shown for Packing Instruction 202 [7.1.5.1(g)]	❑	❑	❑
41. Limited Quantity packagings marked: "LIMITED QUANTITY" or "LTD. QTY." [7.1.5.3]	❑	❑	❑
42. Salvage Packagings marked: "Salvage" [7.1.5.4]	❑	❑	❑
Labelling			
43. The label(s) identifying the Primary risk, affixed to each package, as per 4.2, Column E [7.2.3.2; 7.2.3.3(b)]	❑	❑	
44. The label(s) identifying the Subsidiary risk next to Primary risk label(s), as per 4.2, Column E [7.2.3.2; 7.2.6.2.3]	❑	❑	❑
45. Cargo Aircraft Only label, on the same surface near the Hazard label(s) [7.2.4.2; 7.2.6.3]	❑	❑	❑
46. "Orientation" labels affixed [7.2.4.4]	❑	❑	❑
47. "Magnetized Material" label affixed [4.2, Column E, 7.2.3.10 and 7.2.4.1]	❑	❑	❑
48. "Cryogenic Liquid" labels affixed [7.2.4.3]	❑	❑	❑
49. "Keep Away From Heat" label affixed [7.2.4.5]	❑	❑	❑
50. All above labels correctly affixed [7.2.6] and all irrelevant marks and labels removed [7.1.1; 7.2.1]	❑	❑	
For Overpacks			
51. Packaging Use markings as required must be clearly visible or reproduced on the outside of the overpack [7.1.4.1]	❑	❑	❑
52. If all markings and labels are not visible, the word "Overpack" marked [7.1.4.2]	❑	❑	❑
53. "Cargo Aircraft Only" restrictions [5.0.1.5.3]	❑	❑	❑
GENERAL			
54. State and Operator variations complied with [2.9]	❑	❑	❑
55. Cargo Aircraft Only shipments, a cargo aircraft operates on all sectors.	❑	❑	❑

Comments: _____

_____

Checked by: _____

Place: _____ Signature: _____

Date: _____ Time: _____

*IF ANY BOX IS CHECKED "NO", DO NOT ACCEPT THE SHIPMENT AND GIVE A DUPLICATE COPY OF THIS COMPLETED FORM TO THE SHIPPER.

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Attachment D
DANGEROUS GOODS CHECKLIST FOR A RADIOACTIVE SHIPMENT
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DANGEROUS GOODS CHECKLIST FOR A RADIOACTIVE SHIPMENT

The recommended checklist appearing on the following pages is intended to verify shipments at origin.

Never accept or refuse a shipment before all items have been checked.

Is the following information correct for each entry?

SHIPPERS DECLARATION FOR DANGEROUS GOODS (DGD)

		YES	NO*	N/A
1.	Two copies in English and in the IATA format [10.8.1.2; 10.8.1.4]	❑	❑	
2.	Full name and address of Shipper [10.8.3.1]	❑	❑	
3.	Full name and address of Consignee [10.8.3.2]	❑	❑	
4.	If the Air Waybill number is not shown, enter it. [10.8.3.3]	❑		
5.	The number of pages shown [10.8.3.4]	❑	❑	
6	The non-applicable Aircraft Type deleted [10.8.3.5]	❑	❑	
7.	If full name of Airport or City of Departure or Destination is not shown, enter it. [10.8.3.6 and 10.8.3.7]	❑		
8.	The word "Non-Radioactive" deleted [10.8.3.8]	❑	❑	
Identification				
Note: *Alternative sequence acceptable 10.8.3.9.1(b).*				
9.	UN Number, preceded by prefix "UN" [10.8.3.9.1(a), Step 1a]	❑	❑	
10.	Proper Shipping Name [10.8.3.9.1(a), Step 2a]	❑	❑	
11.	Class 7 [10.8.3.9.1(a), Step 3a]	❑	❑	
12.	Subsidiary Risk, in parentheses, immediately following Class [10.8.3.9.1(a), Step 3a]	❑	❑	❑
13.	Packing Group if required for Subsidiary Risk [10.8.3.9.1(a), Step 4a]	❑	❑	❑
Quantity and Type of Packing				
14.	Name or Symbol of Radionuclide(s) [10.8.3.9.2, Step 5 (a)]	❑	❑	
15.	A description of the physical and chemical form, Special Form, or low dispersible material [10.8.3.9.2, Step 5 (b)]	❑	❑	
16.	The number and type of packages and the activity in becquerel or multiples thereof in each package. For Fissile Material the total weight in grams or kilograms of fissile material may be shown in place of activity [10.8.3.9.2, Step 6]	❑	❑	
17.	For different individual radionuclides, the activity of each radionuclide and the words "All packed in one" [10.8.3.9.2, Step 6]	❑	❑	❑
18.	Activity within limits for Type A packages [Table 10.4.A], Type B, or Type C (see attached competent authority certificate)	❑	❑	❑
19.	Words "overpack used" shown on the DGD [10.8.3.9.2, Step 7]	❑	❑	❑
Packing Instructions				
20.	Category of package(s) or overpack [10.8.3.9.3, Step 8]	❑	❑	
21.	Transport Index and dimensions for Category II and Category III only [10.8.3.9.3, Step 8]	❑	❑	❑
22.	For Fissile Material the Criticality Safety Index [10.8.3.9.3, Step 8]	❑	❑	❑
23.	"Fissile Excepted" [10.8.3.9.3, Step 8]	❑	❑	❑
Authorizations				
24.	Identification marks shown and a copy of the document in English attached to DGD for the following [10.8.3.9.4, Step 9; 10.10.3.1]:			
	– Special Form approval certificate	❑	❑	❑
	– Low dispersible material approval certificate	❑	❑	❑
	– Type B package design approval certificate	❑	❑	❑
	– Type B (M) package shipment approval certificate	❑	❑	❑
	– Type C package design and shipment approval	❑	❑	❑
	– Fissile material package design and shipment approval certificate	❑	❑	❑
	– Special Arrangement shipment approval certificate	❑	❑	❑
25.	**Additional Handling Information** [10.8.3.11]	❑	❑	❑
26.	The air transport statement included or added following Certification Statement [10.8.3.12]	❑	❑	❑
27.	**Name and Title of Signatory, Place and Date** indicated [10.8.3.13 and 10.8.3.14]	❑	❑	

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Attachment D
DANGEROUS GOODS CHECKLIST FOR A RADIOACTIVE SHIPMENT
Page 2 of 2

		YES	NO*	N/A
28.	**Signature** of Shipper [10.8.3.15]	❑	❑	
29.	**Amendment** or alteration signed by Shipper [10.8.1.7]	❑	❑	❑
AIR WAYBILL				
30.	The **Handling Information** box shows: "Dangerous goods as per attached Shipper's Declaration" or "Dangerous Goods as per attached DGD" [10.8.8.1(a)]	❑	❑	
31.	Cargo Aircraft Only or CAO, if applicable [10.8.8.1(b)]	❑	❑	❑
32.	Where non-dangerous goods are included, the number of pieces of dangerous goods shown [10.8.8.2]	❑	❑	❑
PACKAGE(S) AND OVERPACKS				
33.	Number and type of packagings and overpacks delivered as shown on DGD	❑	❑	
34.	Unbroken transportation seal [10.6.2.1.1.2; 10.6.2.2.1] and package in proper condition for carriage [9.1.1.3; 9.1.1.4]	❑	❑	
Markings				
35.	Industrial packages, marked as per 10.7.1.3.3	❑	❑	❑
36.	Type A packages, marked as per 10.7.1.3.4	❑	❑	❑
37.	Type B packages, marked as per 10.7.1.3.5	❑	❑	❑
38.	Type C packages, marked as per 10.7.1.3.6	❑	❑	❑
39	Packages containing Fissile material, marked as per 10.7.1.3.7	❑	❑	❑
40.	The Proper Shipping Name and UN Number [10.7.1.3.1]	❑	❑	
41.	The full Name and Address of the Shipper and Consignee [10.7.1.3.1]	❑	❑	
42.	The permissible gross weight if it exceeds 50 kg [10.7.1.3.1]	❑	❑	❑
Labeling				
43.	Two correctly completed Radioactive Hazard labels on opposite sides [10.7.3.3; 10.7.4.3.1]	❑	❑	
44.	Applicable label(s) identifying the Subsidiary risk next to the Radioactive Hazard labels [10.7.3.2; 10.7.4.3] completed.	❑	❑	❑
45.	Two Cargo Aircraft Only labels, if required, on the same surface near the Hazard labels [10.7.4.2.4; 10.7.4.3.1; 10.7.4.4.1]	❑	❑	❑
46.	For fissile materials, two correctly completed Criticality Safety Index (CSI) labels on the same surface as the hazard labels [10.7.3.3.4; 10.7.4.3.1]	❑	❑	❑
47.	All labels correctly affixed and irrelevant marks and labels removed [10.7.1.1; 10.7.2.1; 10.7.4]	❑	❑	
For Overpacks				
48.	Packaging markings as required must be clearly visible or reproduced on the outside of the overpack [10.7.1.4.1]	❑	❑	❑
49.	If all markings and labels are not visible, the word "Overpack" marked [10.7.1.4.2]	❑	❑	❑
50.	Conditions of 10.7.3.4 complied with	❑	❑	❑
GENERAL				
51.	State and Operator variations complied with [2.9]	❑	❑	❑
52.	Cargo Aircraft Only shipments, a cargo aircraft operates on all sectors	❑	❑	❑
53.	Packages containing Carbon dioxide solid (dry ice), the marking, labelling and documentary requirements complied with [Packing Instruction 904; 7.1.5.1 (d); 7.2.3.9]	❑	❑	❑

Comments: _____

_____

Checked by: _____

Place: _____ Signature: _____

Date: _____ Time: _____

*IF ANY BOX IS CHECKED "NO", DO NOT ACCEPT THE SHIPMENT AND GIVE A DUPLICATE COPY OF THIS COMPLETED FORM TO THE SHIPPER.

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Form WSD-F-0059
ACCEPTANCE OF SAMPLE MATERIALS
Page 1 of 1

This is only an example of the form. See Document Control Center for a usable form.

PADUCAH
Remediation Services
A Portage Shaw Joint Venture Company

Acceptance of Sample Materials

To:_____
Fax number

_____
Contact

_____

Based on attached data and proposed proper shipping name, please confirm the following:

a) _____ This lab can accept the material based on reviewing the radioactive analysis/summary
b) _____ This lab has no administrative action of license revocation pending against it.

NRC Radioactive License No. _____Expiration Date:_____

Signature verification:_____Date_____

Fax back to:
Attn:

Proposed Ship Date: _____

Shipment Doc #:_____

- ☐ TSCA regulated PCB's >500 ppm source
- ☐ RCRA codes attached
- ☐ DOT/IATA-ICAO HM Shipping documents attached
- ☐ Low Specific Activity I Radioactive Material UN2912
- ☐ Non Regulated for Shipment
- ☐ Limited Quantity Radioactive Material UN2910

Page 1 of _____

WSD-F-0059

SAMPLE FORM

Form WSD-F-0060
SAMPLE SHIPMENT PREPARATION CHECKLIST
Page 1 of 1

This is only an example of the form. See Document Control Center for a usable form.

Sample Shipment Preparation Checklist

Sample Shipment Information

1. Document Identification/Reference No.:_____
2. Brief Description of material:_____
3. Proposed shipping date: _____
4. Receiving Facility Name:_____
5. Selected Transporter Name:_____
6. Documents to include in Shipment File:

   a. ____ Draft copy of all shipping documents (Air Waybill, DG Shipper's Dec, DOT BOL, etc.)

   b. ____ Certification of no administrative action or license revocation pending against the receiving facility

   c. ____ Certification that the receiving facility can accept the material

   d. ____ Calculations and Peer Review signature for Hazardous and Non Hazardous Shipments

   e. ____ Appropriate Checklist for Hazardous shipments – must include Peer Reviewer's Signature [Circle One: LTD QTY, IATA Non-Rad, IATA Rad]

   f. ____ Analytical data

   g. ____ HP Survey for Off-Site Release of Samples and Final Package

   h. ____ Confirmation of Sample Delivery (i.e. Fed Ex Tracking Report)

Additional Comments:

_____
_____
_____
_____
_____
_____

WSD-F-0060

SAMPLE FORM

Form WSD-F-0061
LIMITED QUANTITY RADIOACTIVE MATERIALS CHECKLIST/PEER REVIEW
Page 1 of 1

This is only an example of the form. See Document Control Center for a usable form.

Limited Quantity Radioactive Materials Checklist / Peer Review

Ship Date: _____ Air Waybill Number: _____

Requirements for Limited Quantity Shipments of Radioactive Material:

- ☐ 0.5 mRem/hr dose on the surface (Actual dose ____)
- ☐ Meet 10.3.7.2 for fissile materials (< 15 grams U-235 etc.)
- ☐ Contamination Levels do not exceed 10.5.3.13
- ☐ RADIOACTIVE marking on the internal surface where visible upon opening (per 10.5.9.4 for limited quantities)
- ☐ 30 cm dose and 1 meter dose reading (site requirement)
- ☐ Excepted Package meets requirements per 49CFR 173.410, and 10.5.3.1 – 10.5.3.12 as applicable
- ► Excepted from marking EXCEPT for items listed in 10.7.1.3.2:
  - ☐ UN#_____
  - ☐ Name and address of consignor AND consignee
  - ☐ Permissible gross weight (if over 50Kg)
- ► Excepted from labeling
- ☐ Excepted from 10.8.8.1 (placing Dangerous Goods as per attached Dangerous Goods Declaration in the handling information box); however, the endorsements from 10.8.8.3.3, 'Radioactive Material, excepted package – limited quantity of material, UN2910' etc. is required in the 'nature and quantity of goods' box of the air waybill as appropriate.

_____ _____
Signature Date

_____ _____
Peer Reviewer Date

NOTE:
No shipper's declaration of dangerous goods is required for excepted packages of radioactive materials. Therefore, the IATA Radioactive and IATA Non-radioactive checklists are not appropriate.

WSD-F-0061

SAMPLE FORM

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SEC's RADCON Alliance

Auxier & Associates
Thermo NUtech
Kelly Scientific
EDi

AD-MCS-7.10	ISSUE DATE: 4-12-02	REV. NO. 1

CALIBRATION OF GENIE-2000-BASED HPGE GAMMA SPECTROSCOPY SYSTEM

WORK INSTRUCTION

Approvals

Joanne Glenn — 4/11/02
Instrumentation Manager — Date

Roberto D. Perri — 4/11/02
Quality Assurance Manager — Date

REVISION LOG		
Revision Number	Description Of Changes	Pages Affected
0	Initial Release	ALL
1	Replaces Calibration and Operation of GENIE-2000-Based HPGe GAMMA Spectroscopy System	ALL

CONTROLLED DOCUMENT
COPY# 28

NOTE: Work instructions are generated by the SEC RADCON Alliance for use by Alliance employees. They are not to be construed as procedures and do not take precedence over Bechtel Jacobs Company, LLC generated documents.

1.0 PURPOSE

The purpose of this work instruction is to detail the appropriate methods for calibration of the Canberra Genie 2000-based high purity germanium (HPGe) gamma spectroscopy system.

2.0 APPLICABILITY

This document addresses gamma spectroscopy performed for Bechtel Jacobs Company, LLC by the SEC RADCON Alliance (Alliance) at Oak Ridge National Laboratory.

3.0 REFERENCES

3.1 Genie 2000 Operations Manual, current version.

3.2 Model S503 PROcount-2000 Manual

4.0 DEFINITIONS/ACRONYMS

4.1 *Cryostat* – a dewar and a vacuum chamber that houses the detector.

4.2 *Calibration Standard* – a NIST traceable radioactive source used for calibration and QC of a detector.

4.3 *DC* – direct current.

4.4 *DSA 1000* – the electronic assembly consisting of analog to digital converter, multi-channel analyzer, amplifier, and HV power supply.

4.5 *HPGe* – High Purity Germanium, which is the detector crystal.

4.6 *HV* – high voltage

4.7 *QC* – quality control

4.8 *RCL* – radiological counting laboratory

4.9 *RCT* – radiological control technician

4.10 *TS* – technical specialist

4.11 *V* – volts

5.0 RESPONSIBILITIES

5.1 Instrumentation Manager ensures:

5.1.1 The implementation of this work instruction.

5.1.2 That the required review of the records generated by this work instruction is completed.

5.1.3 That Radiological Counting Laboratory (RCL) Radiological Control Technicians are trained to perform this work instruction.

5.1.4 That a copy of this work instruction and reference document(s) are available in the RCL.

5.2 RCL RCT:

5.2.1 Performs calibration of the instrument.

5.2.2 Completes required documentation

5.3 RCL Technical Specialist (TS):

5.3.1 May perform calibration of the instrument.

5.3.2 May complete the required documentation.

5.3.3 Reviews all calibration documentation.

6.0 DETAILED INSTRUCTIONS

6.1 Cautions

6.1.1 High Voltage (HV) Germanium detectors operate at bias voltages up to 5000 V DC. Ensure each detector is grounded through the HV coaxial cable grounding to a grounded power supply/ nuclear instrument module bin.

6.1.2 Caution shall be used when adjusting internal DSA-1000 controls (e.g., to change the polarity of the high voltage power supply). The DSA-1000 shall be disconnected from the power supply before the cover is removed. The DSA-1000 contains capacitors that are a shock hazard, as they may retain charge after being disconnected from the power supply.

6.1.3 Vacuum failure - When a cryostat exhibits signs of catastrophic vacuum failure, such as heavy moisture, ice formation on the surface, or extremely high liquid nitrogen loss rate, stop using the unit immediately, turn the high voltage off and do not allow the unit to warm up. If the unit (detector) is in a shield, close the shield door. Consult the vendor (e.g., www.Canberra.com or at (800) 642-2346).

6.1.4 Radioactive materials – Standards containing radioactive materials may pose an internal and/or external exposure hazard. Individuals performing calibration of the system shall be trained in the proper handling of radioactive materials and the methods of maintaining personal exposures As Low As Reasonably Achievable (ALARA).

6.2 Materials and Equipment

6.2.1 NIST traceable mixed gamma standard;

6.2.2 Gamma Spectroscopy System, including: PC with Genie-2000 spectroscopy software with PROcount and QA options, high purity germanium detector crystal, dewar and detector shield;

6.2.3 Genie 2000 Operation Manual;

6.2.4 Model S503 PROcount-2000 Manual;

6.2.5 Personal protective equipment for handling liquid nitrogen: Face shield, cryogenic gloves, and lab coat.

6.3 Calibration

6.3.1 Full calibration consists of energy calibration, peak shape calibration, and efficiency calibration.

6.3.2 Full calibration shall occur annually or when the system has been down for repair/maintenance or when indicated by daily QC checks. Partial calibration, for energy only, may be performed when indicated by daily QC checks.

6.3.3 Calibrations shall be done by the procedure given in the Genie 2000 Operations Manual.

6.4 Energy Calibration

6.4.1 Energy calibration is performed after repair/maintenance or when indicated by Daily QC Check.

6.4.2 Partial calibration shall be done by the procedure given in the Genie 2000 Operations Manual.

6.5 Instrument Background

6.5.1 Instrument Backgrounds shall be performed annually (at a minimum), or when determined by poor QC Background Check statistics, noticeable constant peaks in sample spectra or site-specific requirements.

6.5.2 Instrument Background shall be determined according to the procedure given in the Genie-2000 Operations Manual.

6.5.3 The suggested minimum time is 16 hours or 57,600 seconds.

7.0 RECORDS

7.1 The following records shall be maintained for the life of the project in accordance with AD-MCS-7.4, *SEC RADCON Alliance Record Management*.

7.1.1 Calibration Reports (see examples, Attachments A, B, C and D)

7.1.2 Calibration Standards Certification (see example, Attachment E).

8.0 ATTACHMENTS

8.1 Attachment A, Example Energy Calibration Report

8.2 Attachment B, Example Efficiency Calibration Report

8.3 Attachment C, Example Energy Verses Channel Graph

8.4 Attachment D, Example Efficiency Calibration Graph

8.5 Attachment E, Example Calibration Standards Certification

Attachment A

Example Energy Calibration Report

```
Energy Calibration Report              12/13/2001  9:12:01 AM       Page  1
*************************************************************************
*****       E N E R G Y   C A L I B R A T I O N   R E P O R T       *****
*************************************************************************
     Detector Name:  DET01
     Sample Title:
       *************  ENERGY CALIBRATION COEFFICIENTS  *************
              Energy Calibrate Performed on:  12/13/2001  9:11:42 AM
                      by: x
              Energy Calibrate Type:          POLY
   Energy(keV) =     0.134 +     0.161*ch +  0.00E+000*ch^2 +  0.00E+000*ch^3

       *************  SHAPE CALIBRATION COEFFICIENTS   *************

              Shape Calibrate Performed on:  11/26/2001  3:21:01 PM
                      by: x

              FWHM      =     0.877  +   0.034*E^1/2

              LOW TAIL =   0.0E+000 +  0.0E+000*E

****************     ENERGY CALIBRATION RESULTS TABLE     ****************
```

Centroid Channel	Centroid error	Energy (keV)
369.47	0.05	60.00
546.26	0.05	88.00
757.18	0.05	122.00
986.12	0.05	159.00
1984.69	0.07	320.00
2428.71	0.04	392.00
3186.86	0.04	514.00
4102.32	0.04	662.00
5568.53	0.03	898.00
7275.52	0.04	1173.00
8263.99	0.04	1333.00

```
*********     SHAPE CALIBRATION RESULTS TABLE     *********
```

Energy (keV)	FWHM channels	FWHM error	TAIL channels	TAIL error
60.00	0.00	0.00	0.00	0.00
88.00	0.00	0.00	0.00	0.00
122.00	0.00	0.00	0.00	0.00
159.00	0.00	0.00	0.00	0.00
320.00	0.00	0.00	0.00	0.00
392.00	0.00	0.00	0.00	0.00
514.00	0.00	0.00	0.00	0.00
662.00	0.00	0.00	0.00	0.00
898.00	0.00	0.00	0.00	0.00
1173.00	0.00	0.00	0.00	0.00
1333.00	0.00	0.00	0.00	0.00

Attachment B
Example Efficiency Calibration Report

```
Efficiency Calibration Report                12/13/2001  9:13:58 AM   Page  1
*******************************************************************************
*********   E F F I C I E N C Y   C A L I B R A T I O N   R E P O R T   *****
*******************************************************************************
     Detector Name:  DET01
     Sample Title:
     Geometry Description: Petri
     Efficiency Calibration Performed on:  12/13/2001  9:12:19 AM
                    by: x
     Geometry Type Used:   DUAL
```

Efficiency Triplets

Energy	Efficiency	Error
60.00	7.03E-002	2.26E-003
88.00	7.58E-002	2.52E-003
122.00	7.07E-002	2.45E-003
159.00	5.91E-002	2.09E-003
320.00	3.61E-002	1.48E-003
392.00	3.09E-002	1.01E-003
514.00	2.47E-002	7.92E-004
662.00	2.10E-002	6.89E-004
898.00	1.55E-002	4.91E-004
1173.00	1.26E-002	3.99E-004
1333.00	1.13E-002	3.59E-004
1836.00	8.96E-003	2.86E-004

DUAL Efficiency Calibration Equation

Single Equation Terms ->	Offset:	-154.826
	Slope:	127.863
	Quadratic:	-42.479
	Cubic:	7.013
	4th Order:	-0.578
	5th Order:	0.019
	6th Order:	0.000
	7th Order:	0.000
	8th Order:	0.000
	9th Order:	0.000

```
Efficiency Calibration Report                12/13/2001  9:13:58 AM   Page  2
```

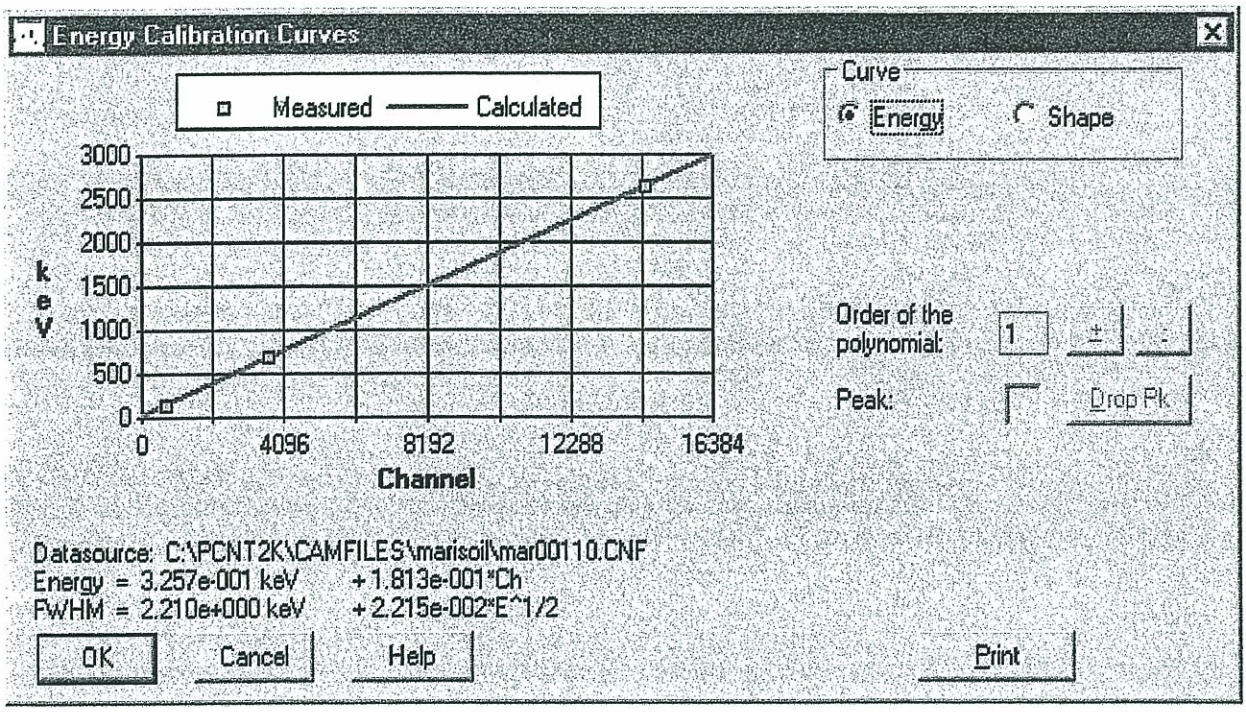
EMPIRICAL Efficiency Calibration Equation

Empirical Equation Terms ->	Scaling:	948.000
	Offset:	-4.197
	Slope:	0.855
	Quadratic:	0.002
	Cubic:	-0.099
	4th Order:	0.074
	5th Order:	-0.019

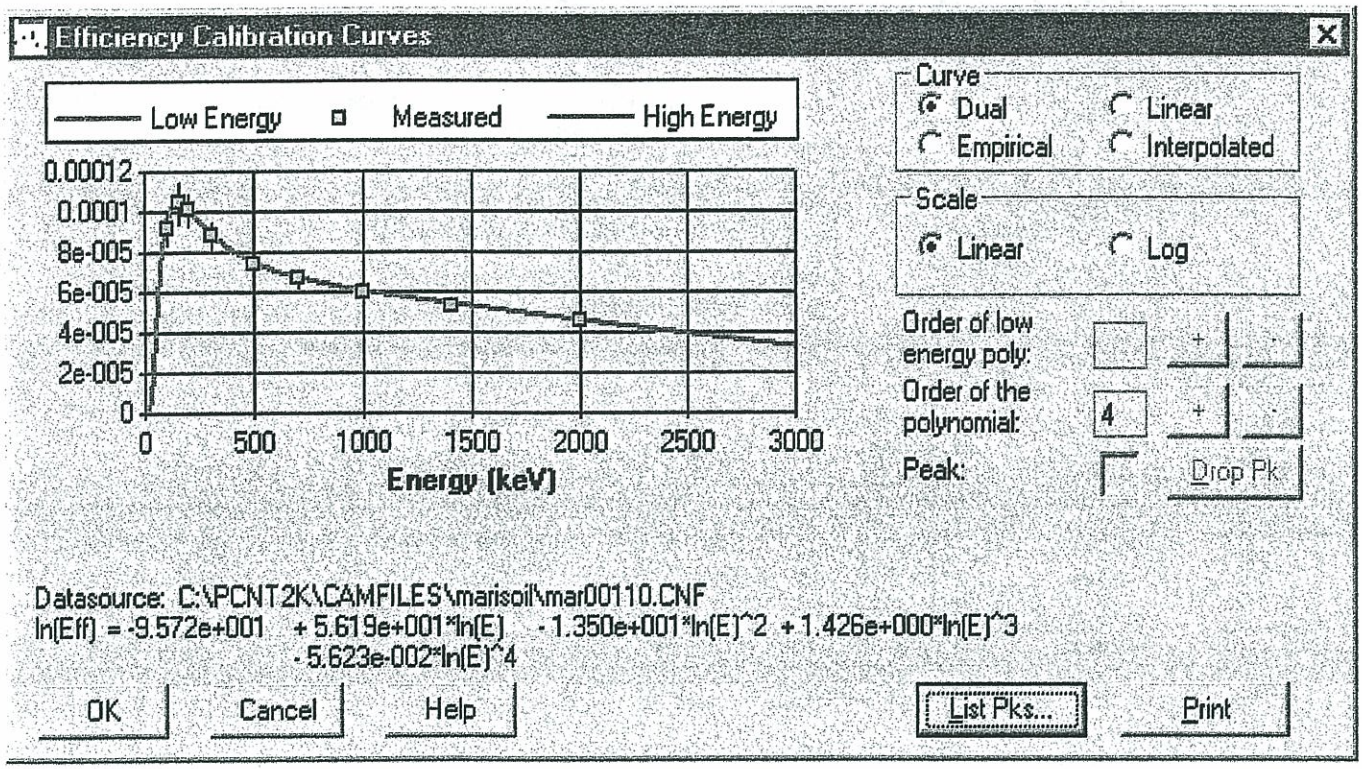
LINEAR Efficiency Calibration Equation

Linear Equation Terms ->	Offset:	-0.000
	Slope:	-1.901
	Quadratic:	243.596
	Cubic:	-30872.340
	4th Order:	1876099.250
	5th Order:	-44242216.0
	6th Order:	0.000
	7th Order:	0.000
	8th Order:	0.000
	9th Order:	0.0

Attachment C
Example Energy versus Channel Graph



Attachment D
Example Efficiency Calibration Graph



Attachment E,
Example Calibration Standards Certificate

Isotope Products Laboratories
An Eckert & Ziegler Company

24937 Avenue Tibbitts
Valencia, California 91355

Tel 661•309•1010
Fax 661•257•8303

CERTIFICATE OF CALIBRATION
MULTINUCLIDE STANDARD SOURCE

Customer SEC (SAFETY AND ECOLOGY CORPORATION)
Catalog No.: EAB-CUST
Source No.: A2-525

P.O. No.: 0002410
Reference Date: 15-Sep-01 12:00 PST
Total Radioactivity: 3.004 µCi
Total Radioactivity: 111.1 kBq

Description of Source:

A. Capsule type: Customer supplied planchet (47 mm OD)
B. Nature of active deposit: Distributed and evaporated metallic salt on filter paper
C. Active diameter/volume: 41 mm
D. Backing: Aluminum
E. Cover: 0.9 mg/cm² aluminized mylar

Gamma-Ray Energy (keV)	Nuclide	Half-life	Branching Ratio (%)	Activity (µCi)	Gammas per second	Total Uncert.
60	Am-241	432.17 ± 0.66 years	36.0	0.08711	1160	3.1%
88	Cd-109	462.6 ± 0.7 days	3.63	0.8441	1134	3.2%
122	Co-57	271.79 ± 0.09 days	85.6	0.03109	984.7	3.3%
159	Te-123m	119.7 ± 0.1 days	84.0	0.03974	1235	3.3%
320	Cr-51	27.706 ± 0.007 days	9.86	1.029	3754	3.1%
392	Sn-113	115.09 ± 0.04 days	64.89	0.1565	3757	3.1%
514	Sr-85	64.849 ± 0.004 days	98.4	0.1994	7260	3.1%
662	Cs-137	30.17 ± 0.16 years	85.1	0.1362	4289	3.2%
898	Y-88	106.630 ± 0.025 days	94.0	0.3160	10990	3.1%
1173	Co-60	5.272 ± 0.001 years	99.86	0.1646	6082	3.1%
1333	Co-60	5.272 ± 0.001 years	99.98	0.1646	6089	3.1%
1836	Y-88	106.630 ± 0.025 days	99.36	0.3160	11620	3.1%

Method of Calibration:
The source was prepared from weighed aliquots of solutions whose concentrations in µCi/g were determined by gamma spectrometry.

Notes:
- See reverse side for leak test(s) applied to this source.
- Nuclear data was taken from IAEA-TECDOC-619, 1991.
- IPL participates in a NIST measurement assurance program to establish and maintain implicit traceability for a number of nuclides, based on the blind assay (and later NIST certification) of Standard Reference Materials (As in NRC Regulatory Guide 4.15).
- Overall uncertainty is calculated at the 99% confidence level.
- This source has a working life of 1 year.

Ann U Khan
Quality Control

26-Sep-01
Date Signed

IPL Ref No.: 752-43

ISO 9001 CERTIFIED

Medical Imaging Laboratory
24937 Avenue Tibbitts Valencia, California 91355

Industrial Gauging Laboratory
1800 North Keystone Street Burbank, California 91504

AD-MCS-7.12	ISSUE DATE: 7/21/05	REV. NO. 2

OPERATION OF GENIE-2000-BASED HPGE GAMMA SPECTROSCOPY SYSTEM

WORK INSTRUCTION

Approvals

Instrumentation Manager — Date: 7/20/05

Quality Assurance Manager — Date: 7/20/05

REVISION LOG		
Revision Number	Description Of Changes	Pages Affected
0	Initial Release	ALL
1	Extensive re-write	ALL
2	Revised to address ISOCS issues	2, 3, 4, 5

NOTE: Work Instructions are generated by the SEC RADCON Alliance for use by Alliance employees. They are not to be construed as procedures and shall not take precedence over Bechtel Jacobs Company LLC generated documents.

1.0 PURPOSE

This procedure establishes the method for the operation, calibration, and maintenance of all HPGe gamma spectroscopy systems. This system is used for the identification of and, where practical, quantification of radionuclides present at the ORNL, Y-12, ETTP, and Paducah facilities. Information derived will be used for assessing the radiological hazards and condition present as well as decision making for worker protection.

2.0 APPLICABILITY

This document addresses all gamma spectroscopy systems operated for Bechtel Jacobs Company, LLC by the SEC RADCON Alliance. Due to the complexity of Insitu Gamma spectroscopy system software and the verity of modeling applications, Insitu operation requires advanced training to address the use of insitu modeling and is maintained on file in the training department.

3.0 REFERENCES

3.1 Canberra Genie System Spectroscopy Applications and Display User's Guides, 07-0196-09, VMS Spectroscopy Applications Package User's Manual, Vol. 1 June 1994.

3.2 Kocher, D. C., "Radioactive Decay Data Tables," DOE/TIC-11026, 1981, available from U. S. National Technical Information Service.

3.3 SEC-QA-101, *SEC RADCON Alliance Quality Assurance Project Plan.*

3.4 AD-MCS-7.4, *SEC RADCON Alliance Record Management.*

3.5 ASTM Standard C 1402-98, Standard Guide for High-Resolution Gamma-Ray Spectroscopy of Soil Samples, available from American National Standards Institute.

3.6 ANSI/IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors, IEEE Std 325-1996.

3.7 Genie 2000 Spectroscopy System Operations 9230846G 3/01

4.0 DEFINITIONS

4.1 *Efficiency* – the percent of decay events from a standard radioactive source, in a specific reproducible geometry, that are seen and measured by a detector.

4.2 *Traceable Calibration Standard* – a calibrated radioactive source prepared as or from a standard reference material traceable to National Institute of Standards & Technology (NIST).

4.3 Check Source – a radioactive source used to confirm the satisfactory operation of the instrument.

4.4 FWHM (Full Width Half Maximum) – the full width of a gamma-ray peak distribution measured at half the maximum peak height, measured above the continuum (background).

4.5 Dead Time – the time while the amplifier is processing a pulse and is unable to process another pulse.

4.6 Geometry – a standard sample or source counting configuration (i.e. Marinelli beaker, Petri dish, and others) and its relationship to the detector.

4.7 ISOCS (In-situ object counting system)- refers to a HPGe gamma spectroscopy systems that has a mathematical detector response characterization that allows for efficiency calibration using a object modeling software.

5.0 RESPONSIBILITIES

5.1 Laboratory Manager (or designee) ensures:

5.1.1 The implementation of this work instruction

5.1.2 That the required review of the records generated by this work instruction is completed.

5.1.3 That Radiological Counting Laboratory (RCL) Radiological Control Technicians (RCT) are trained to perform this work instruction.

5.1.4 That a copy of this work instruction and reference document(s) are available in the RCL.

5.2 Radiological Control Technicians:

5.2.1 Operates the instrument as required in this work instruction.

5.2.2 Completes the required documentation prescribed in this work instruction.

5.3 Technical Specialist (TS)

5.3.1 Reviews data for technical accuracy.

5.3.2 Aids in troubleshooting.

6.0 DETAILED INSTRUCTIONS

6.1 Cautions

6.1.1 Individuals performing this procedure should be aware of the precautions necessary for the proper handling of radioactive materials, as defined the Activity Hazard Analysis (AHA) and corresponding Radiological Work Permit (RWP)

6.1.2 Follow the manufacturer's instructions for set up, inter-component connections, and preliminary testing of the equipment. Observe all of the manufacturer's limitations and cautions.

6.1.3 When handling LN_2 (liquid nitrogen), stay clear of confined spaces, wear non-absorbent, insulated gloves, face shield, and observe all compressed gas safety precautions found on the Material Safety Data Sheets and as defined in the AHA.

6.2 Interferences

There are radionuclides that emit gamma rays at similar energies to other radionuclides. The nuclide library should be designed to limit the impact of radionuclides that emit gamma rays at similar energies. Additionally, the library

should be customized to incorporate only the radionuclides known to exist. The radionuclides present are determined from the site history. Nuclide libraries (RADCON.lib) for routine sample analyses have already been developed for this procedure and should not be altered without the Laboratory Manager's approval. Other libraries may be used on a case-by-case basis for non-routine purposes, and analytical results derived from these libraries require scrutiny.

6.3 Materials and Equipment

6.3.1 High Purity Germanium detector with built in preamplifier

6.3.2 Graded lead shield

6.3.3 Dewar flask (5-30 Liter)

6.3.4 6.2.4 DSA -1000 (integrated multi-channel analyzer) or equivalent

6.3.5 6.2.5 Personal computer workstation, or equivalent, with gamma analysis and QA software

6.3.6 Electronic Balance and Weight Set

6.3.7 Associated sample containers/ plastic bags

6.4 Quality Control

6.4.1 Efficiency calibration will be performed for all counting geometries with a NIST traceable calibration standard that closely approximates the sample counting geometry when defensible quantitative results are required.

6.4.2 The Laboratory Manager (or designee) will review all data generated by this procedure.

6.4.3 The calibration portions of this procedure will be performed at initial installation, following system maintenance that may affect the electronics, when a problem is suspected, and at the following frequencies.

6.4.4 Efficiency Calibration – performed annually or as needed.

6.4.5 ISOCS Characterization- initial performed by the manufacture and as required after any repair's preformed by the manufacture that effect's the detector's characterization.

6.4.6 Energy and FWHM Calibration – performed annually or as needed.

6.4.7 The operator performing this procedure will ensure that all daily instrument checks have been performed and meet performance criteria before counting any samples.

6.4.8 Detector backgrounds subtraction files will be established at least once each calendar week for system where background subtraction is used

6.4.9 Data will be backed up on long-term storage media (i.e. compact disk) on a monthly basis. This data backup is to include all sample spectrum files (filename.cnf) and the entire directory C:\program files\Procount.

6.5 Filling Dewars with Liquid Nitrogen

6.5.1 For HPGe (High Purity Germanium) detectors, the crystal may be allowed to warm to room temperature and subsequently cooled. These cycles should be minimized to extend the life of the crystal. If warmed, the

detector must be warmed all the way to room temperature before cooling. Remove the cryostat from liquid nitrogen for 24 hours before attempting to cool the detector. For HPGe system that have an integral dewar, place the unit on it's side to allow the liquid nitrogen to drain from the dewar.

6.5.2 Ensure the detector cold-finger is submersed in liquid nitrogen at least 12 hours before applying a bias to the crystal or as recommended by the manufacturer of that detector.

CAUTION: Compressed gas and temperature hazard (see Sec. 6.1.3).

6.5.3 The high purity germanium detectors used for measuring gamma ray emissions are designed to operate at very low (cryogenic) temperatures. These temperatures are attained by immersing the cold finger or dipstick of the detector in a dewar flask containing a cryogenic fluid (liquid nitrogen). The following method describes the proper procedure for filling the dewar flasks with liquid nitrogen. This procedure will be performed on a weekly basis, with intervals between fillings not to exceed eight (8) days.

6.5.3.1 Ensure all detector fill vent and the vent line drain valve are open.

6.5.3.2 Verify the liquid nitrogen supply line is connected to the fill tube of the detector dewar. If it is not, connect it.

6.5.3.3 Open the supply valve on the supply dewar.

6.5.3.4 When liquid begins to flow from the overflow line, shut the fill valve and record the detector number and date the detector dewar was filled in the Liquid Nitrogen Fill Log (see attachment A).

6.6 Initial Detector setup

6.6.1 The gamma spectroscopy system is initially configured and installed by the manufacturer. See the Germanium Detectors User's Manual designed for each detector for a description on how to assemble the detector system.

6.6.2 The high voltage power supply must be matched to the detector see the Germanium Detectors User's Manual designed for each detector for a description on the required high voltage settings. Set the high voltage power supply to these settings, instructions on making these settings can be found in the manufacturer's manual.

6.6.3 The amplifier must be setup to match the detector. See the Germanium Detectors User's Manual designed for each detector for a description on the required amplifier settings. Set the amplifier to these settings, instructions on making these settings can be found in the manufacturer's manual.

6.6.4 The ADC must be setup according to the manufacturer's instructions. Set the ADC for an 8192-channel spectrum.

6.7 Front End Electronic Setup

6.7.1 Ensure the high voltage power supply is set to zero before energizing the unit. Turn on the high voltage power supply and begin to slowly raise the

voltage (approximately 100 V/sec) until the correct operating voltage is reached. Never exceed the manufacturer's recommended operating voltage for the detector.

6.7.2 The amplifier has a pole-zero adjustment that must be fine-tuned in order to maximize detector resolution and obtain correct peak shape. Follow the detailed instructions given in Attachment B, Genie System Spectroscopy Applications and Display User's Guides 07-0196-09 Volume 1, Appendix B.

6.7.3 Adjust the ADC ZERO and Lower Level Discriminator (LLD) as described in Attachment B, Genie System Spectroscopy Applications and Display User's Guides 07-0196-09 Volume 1, Appendix B.

6.7.4 Perform the analog energy calibration as described in Attachment B, Genie System Spectroscopy Applications and Display User's Guides 07-0196-09 Volume 1, Appendix B. The desired energy calibration is 0.25 keV per channel.

6.8 Energy and FWHM (Shape) Calibration

6.8.1 A certificate file must be created that contains the decay date and gamma ray information from the NIST traceable calibration standard.

Note: The volume entered in the certificate file must always be one (1).

6.8.2 Energy and shape calibration shall be performed either during initial instrument setup, annually, after system maintenance that may affect the calibration, prior to efficiency calibrating, or whenever a problem is suspected.

6.8.3 Position an appropriate NIST traceable calibration standard on the detector and count until a minimum of 10,000 counts has been acquired in each of the target peaks.

6.8.4 Review the calibration results displayed for energy and FWHM. The results for FWHM should be compared to the manufacturer specifications for that detector. Differences in the setup of the electronics can lead to slight variances in the manufacturer's rated FWHM versus the actual FWHM values. A detailed explanation of FWHM and its calculation can be found in Reference 13.4 ANSI/IEEE Standard Test Procedures for Germanium Gamma-Ray Detectors.

6.8.5 The energy calibration from the geometry that is used most often will be applied to all geometries.

6.8.6 The energy calibration plot shall be a straight line, which reflects the linearity of the calibration (i.e. 0.25 keV per channel).

6.8.7 Submit the calibration report to the Laboratory Manager for review.

6.9 Efficiency Calibration

6.9.1 A certificate file must be created that contains the decay date and gamma ray information from the NIST traceable calibration standard.

6.9.2 Efficiency calibration shall be performed either during initial instrument setup, after system maintenance that may have affected the calibration, or annually.

6.9.3 The NIST traceable calibration standard shall cover the entire range of energies used in the counting libraries.

6.9.4 The NIST traceable calibration standard should have a matrix and/or density similar to that of the samples so that the attenuation can be closely represented. Each sample matrix should have a different NIST traceable calibration standard (Examples: soil, water, and air filter paper).

6.9.5 Position an appropriate NIST traceable calibration standard on the detector and count until a minimum of 10,000 counts has been acquired in each of the target peaks.

6.9.6 The efficiency calibration software shall determine the appropriate efficiency selection by using the reduced chi-squared statistic as a goodness-of-fit indicator (e.g. Dual efficiency). The chi-squared value should be close to one.

6.9.7 Submit the efficiency calibration report to the Laboratory Manager for review.

6.10 Daily Quality Control Checks

6.10.1 The daily quality control (QC) performance check shall be performed daily prior to counting samples, following instrument maintenance which may affect the system calibration, or whenever a problem is suspected.

6.10.2 Position the appropriate NIST traceable calibration/check source on the detector in the proper position and start data acquisition.

6.10.3 After completion of data acquisition, a daily QC report is automatically generated. The predetermined parameters (i.e., CS-137 activity, CS-137 peak centroid, Cs-137 efficiency, etc.) are checked to verify that the results are within acceptable limits. A check source should have gamma peaks that cover the range (i.e., low, mid, high) of detection, generally 59 keV to 2000 keV. If a parameter is outside acceptable limits, a flag (i.e., investigate, 2-sigma; action, 3-sigma) will be printed on the Daily QC Report. If any of the parameters indicate an Action flag, contact the Laboratory Manager, investigate the cause, and recount the standard. If any parameters indicate an action flag a second time, contact the Laboratory Manager and proceed under his/her direction.

6.11 Daily Background Check

6.11.1 The daily background check shall be performed daily prior to counting samples. This check is performed to verify that the detector is not contaminated.

6.11.2 Check the detector chamber to verify that it is empty and close the lid.

6.11.3 Start acquisition of the daily background check for 1000 seconds.

6.11.4 After completion of data acquisition, a daily background report is automatically generated. The predetermined parameter (i.e., background count rate) is checked to verify that the results are within acceptable limits. If the parameter is outside acceptable limits, a flag (i.e., investigate, 2-sigma; action, 3-sigma) will be printed on the Daily Background Report. If the background rate parameter indicates an Action flag, contact the Laboratory Manager, investigate the cause, and recount the empty chamber. If any parameters indicate an action flag a second time, contact the Laboratory Manager and proceed under his/her direction.

6.12 Weekly Chamber Background Count

6.12.1 The weekly chamber background count shall be performed weekly for a predetermined count time. The predetermined background count time must be equal to or greater than the sample count time. The weekly background count is used for background subtraction in the activity calculation. An accurate representation of the background for the detector is necessary to produce high quality sample results.

6.12.2 Ensure that the detector chamber is empty and close the lid.

6.12.3 Start acquisition of the weekly background count for 57600 seconds.

6.12.4 After completion of data acquisition, a weekly background report is automatically generated. If any problems are discovered (i.e., sample left on the detector) contact the Laboratory Manager and proceed under his/her direction.

6.13 Sample Receiving

6.13.1 All samples received shall be logged into the "Sample Accountability Log" (See Attachment C) and assigned a sample number.

6.13.2 Samples submitted to the laboratory should be under the direction of the Project Health Physicist (PHP) or Radiological Control Technician Supervisor (Lead RCT).

6.14 Sample Preparation

6.14.1 All sample preparation should be performed as per the site-specific radiological work permit (RWP).

6.14.2 A calibrated scale will be used to determine all weights. The scale shall be performance checked weekly prior to use. This weekly performance check will consist of checking the scale response using three weights that span the range of normal operation (110g, 500g, 2000g). Results of the weekly performance check shall be recorded in the Scale Calibration Log (See Attachment D).

6.14.3 Weigh an empty Marinelli or Petri dish (i.e., tare weight) and then fill with the required amount of sample. Close the sample container and secure the lid of the Marinelli or Petri dish with electrical tape. Determine net weight of the sample then record on the Marinelli or Petri dish. Place labeled Marinelli beaker or Petri dish in plastic bag. Inspect for any leakage before submitting for gamma spectroscopy

6.15 Sample Counting

6.15.1 Ensure the daily quality control check and daily background check have been performed and all parameters are in bounds.

6.15.2 Ensure the sample is in a standard calibrated geometry and is in a new plastic bag.

6.15.3 If a standard calibrated geometry is not possible due to sample matrix, the results should be marked as qualitative only.

6.15.4 Record the required sample information in the Gamma Spectroscopy Run Log (see Attachment E).

6.15.5 Place the sample on the detector and close the lid.

CAUTION: Be careful of pinch points when closing lead lid.

6.15.6 Enter the sample information from the Gamma Spectroscopy Run Log and start data acquisition. Select the count time based upon the field screening information, sample origin and matrix. (e.g., water samples 16 hr., soil sample 1hr., and filter samples 8 hr.)

6.15.7 After completion of data acquisition, a raw data report is automatically generated. This raw data report shall be kept within a file folder until completion of twenty samples or the end of the month. Additionally a text file containing the sample results is automatically generated and stored by sample id in the directory C:\ Procount\Repfiles. For immediate notification, this file is to be e-mailed to the requester upon completion of counting and preliminary data review.

6.15.8 The preliminary data review, at a minimum will include a review of the hard copy data report to check for consistence with the Sample Accountability Log. Technical accuracy to check that the identified isotopes are indeed present and a reasonable attempt has been made to determine the identity of all unidentified peak that are significantly above the base line.

6.16 Sample Disposal/Return

All residual sample matrices, including soil, water, filters, etc., are required to be picked-up by the individual submitting the samples for analysis. If samples require transportation on public roads (return to Y-12) they are either segregated at the time of their analysis to ensure that these residual samples, when packaged for transport, will not exceed the DOT's activity limit of 70 Bq/g or will be appropriately labeled as described in AD-MCS-7.1, Hazardous and Radioactive Materials Transportation Work Instruction.

7.0 RECORDS

7.1 Data Package Generation and Transmittal

7.1.1 Assemble all hard copy raw data files specific to the group, including sample accountability logs, balance check logs, and run logs in an orderly fashion contained into a single folder or file.

7.1.2 Generate a results summary using the automated program in excel titled AutoPC.XLS. Place all raw data files (filename.rpt) into the desktop

folder titled "raw data", open the AutoPC.xls program and press the button titled "get data" and "make reports." Follow all on screen directions

7.1.3 Print a copy of the report (results summary) and include it in the data package.

7.1.4 Generate a case narrative; include this as the cover of the completed data package.

7.1.5 This data package is to be submitted to the Alliance Records Specialist and retained as a QA record in accordance with reference 3.4

7.2 Monthly Quality Control Package

The daily quality control samples shall be plotted to indicate trends or other problems. These control charts shall be assembled into a package that contains all the control charts, source certificates, energy and efficiency calibrations, maintenance logs, and a narrative that explains any quality failures or trends. Additionally this package will include sample throughput records. This package is to be assembled once per month and submitted to the Project Health Physicist (PHP) for approval. Forward the complete and approved QA package to the Alliance Records Specialist and retain as a QA record in accordance with reference 3.4

8.0 ATTACHMENTS

8.1 Attachment A, Liquid Nitrogen Fill Log.

8.2 Attachment B, Genie System Spectroscopy Applications and Display User's Guides 07-0196-09 Volume 1, Appendix B.

8.3 Attachment C, Sample Accountability Log

8.4 Attachment D, Scale Calibration Log

8.5 Attachment E, Gamma Spectroscopy Run Log

Attachment A

Liquid Nitrogen Fill Log

DATE FILLED	INITIALS

DATE FILLED	INITIALS

Attachment B

Genie System Spectroscopy Applications and Display User's Guides 07-0196-09 Volume 1

APPENDIX B

AMPLIFIER/ADC SET UP PROCEDURES

B.1 Amplifier Pole-Zero Adjustment

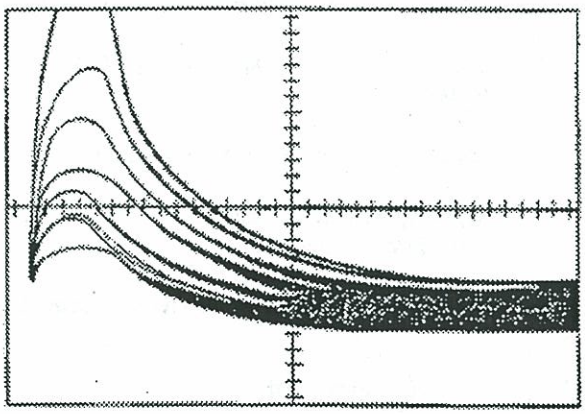
Each amplifier has its own pole-zero adjustment. The description here is for a typical nuclear spectroscopy amplifier. For more specific details, refer to instructions supplied by the manufacturer of the amplifier.

The pole-zero circuit controls the pulse shape generated by the amplifier. To monitor the pulse shape you must have an amplifier, a detector/preamplifier, a gamma-ray source, and an oscilloscope. Everything must be powered up, and the source must be placed near the detector. Create an MCA configuration to take data from the ADC.

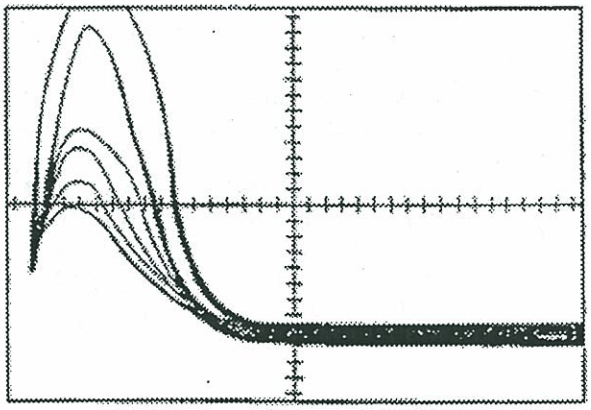
1. Route the amplifier's unipolar output to the oscilloscope. As a starting point, set the oscilloscope up for 2 microsecond/cm sweep and 0.5 v/cm vertical scale. Adjust the pulse height with the amplifier's gain controls (and the oscilloscope controls) so that the display is contained entirely on the screen. The display on the screen should look like one of the figures on the next page. The highest pulses should reach at least several volts (but no higher than 10 volts).

2. Adjust the pole-zero set screw back and forth until the pole-zero circuit is properly adjusted, as illustrated in the middle figure: the baseline portion of the waveform should be as narrow as possible.

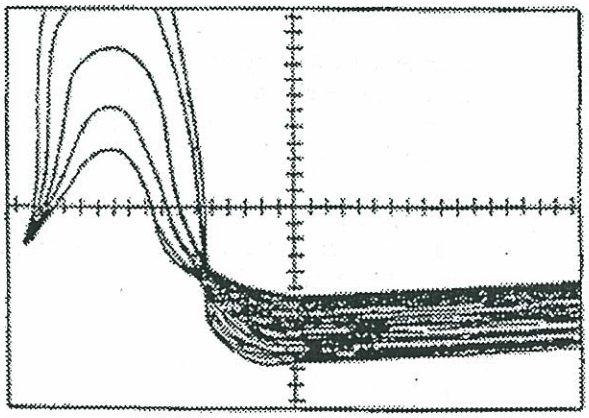
AMPLIFIER/ADC SET UP PROCEDURES
AMPLIFIER POLE-ZERO ADJUSTMENT



Undershoot



Correct



Overshoot

B.2 ADC Analog Zero Adjustment

The following discussion assumes that an ND580 series ADC is being adjusted.

1. Set the ADC acquire mode switch to STROBE and the coincidence mode switch to OFF.

2. Erase the spectrum. Expand the spectral display so that only the first few hundred channels are seen.

3. Disconnect the amplifier from the ADC.

4. Start acquisition. Adjust the ADC ZERO control until storage of strobe pulses is observed in the first channel.

Note that some older amplifiers may also require an analog zero adjustment.

B.3 LLD Adjustment

1. Set the ADC mode switch to ACQUIRE and display the entire spectrum. Place a source near the detector, erase, and start acquisition.

2. You may have to adjust the LLD control upward until storage of a spectrum is observed (if the LLD is initially adjusted too low, the ADC may lock up with 100 percent dead time).

3. Generally, it is desirable to set the LLD as low as possible without increasing system deadtime. The Busy meter on the front panel of the ADC may be used to determine the proper setting. Turn the LLD down (counterclockwise) slowly until the Busy sharply increases. Then turn the LLD back up until the Busy returns to normal.

B.4 Analog Energy Calibration

1. Determine how many keV per channel are desired: divide the highest energy desired by the number of channels in the spectrum. For example, a 4096 channel spectrum, with an energy range between 0 and approximately 2 MeV, is typically setup to have 0.5 keV per channel.

2. Obtain a source which has a peaks at both a low energy and at a high energy. (For example, a composite CO-60/CO-57 source has peaks at 122 and 1332 keV.) Calculate the channel numbers at which these peaks should appear. (For the example peaks above, these will be 244 and 2664.)

3. Begin acquiring data with the source. Place the cursor at the channel for the high energy peak. Adjust the amplifier gain controls until the peak is acquiring at that channel.

4. Move the cursor to the channel for the low energy peak. The peak should be there. If not, adjust the ADC ZERO until it is. Then repeat these last two steps.

5. Readjust the ADC LLD setting.

At this point, the front end is set up correctly. You must still determine the exact energy calibration factors via the CALIBRATE command (see the section on Calibration in Chapter 2).

Attachment C
Example Sample Accountability Log

Revision 0
March 22, 2002

Form IS-437-001

Sample Accountability Log

Sample Code: (1) Smear; (2) Air sample; (3) Gamma Spec.

RADCON Area	Name of Deliverer	Work Release #	Charge #	Date/Time Relinquished Date	Time	Site	Sample Type Code	Bldg#	Sample id (If sample code #2, Then enter web survey sample id#))	** Meets RWP * see foot note (Y/N)

(1) Package Exterior
<20 dpm/100cm^2(α)
<200dpm/100cm^2(βγ)

(2) Package contents
<2K dpm/100cm^2 (α)
<20K dpm/100cm^2(βγ)

(3) Dose Rate < 5mR/hr@30cm

Attachment D
Scale Calibration Log

DATE	INSTRUMENT ID	KNOWN VALUE	MEASURED VALUE	WITHIN QC LIMITS	INITIALS

Attachment E
Gamma Spectroscopy Run Log

SAMPLE ID	FILE NUMBER	COUNT TIME	MATRIX	WEIGHT	SAMPLE DATE	SAMPLE TIME	ACQUISITION DATE	ACQUISITION TIME	SITE WBS#	ANALYST

PCB (Polychlorinated Biphenyls)

Immunoassay Method*

For Soil and Water

This method analyzes for PCB that has been extracted from soil samples. Sample extracts, calibrators, and reagents are added to cuvettes coated with PCB-specific antibodies. The color that develops is then measured and compared with the color measurements of the calibrators. The test requires about 20 minutes for complete analysis. As many as 10 cuvettes can be run simultaneously.

Tips and Techniques

- **Read the entire procedure before starting.** Identify and have ready all the necessary reagents, cuvettes, and other apparatus before beginning the analysis.
- **Timing is critical**; follow instructions carefully.
- **A consistent technique when mixing the cuvettes is critical to this test.** The best results come from using the cuvette rack and mixing as described in *Using the 1-cm MicroCuvette Rack.* Cuvettes can be mixed individually, but test results may not be as consistent.
- Handle the cuvettes carefully. Scratches on the inside or outside may cause erroneous results. Carefully clean the outside of the cuvettes with a clean absorbent cloth or tissue before placing them into the instrument. (Kimwipe® tissues are provided with the kit.)
- Antibody cuvettes and enzyme conjugate are made in matched lots. Do not mix reagent lots.
- Twenty Antibody Cuvettes are provided with each reagent set. One Antibody Cuvette will be used for each calibrator or sample. Antibody Cuvettes are not reusable.
- To avoid damaging the Color Developing Solution, do not expose it to direct sunlight.
- There are two protocols in this procedure, one for levels of 1 ppm and 5 ppm, and another for 10 ppm and 50 ppm. Each uses a different quantity of calibrator and sample extract as follows:

Range (as Arochlor 1248)	Volume of calibrator and sample extract used
1 ppm and 5 ppm	50 µL
10 ppm and 50 ppm	10 µL

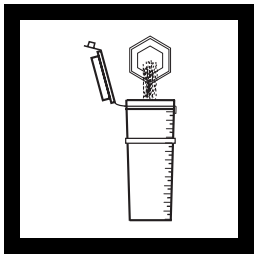
- To test across ranges, such as 1 and 50 ppm, test the lower concentration first. If the result is positive then test at the higher level. If the result of the test at the lower concentration is negative, the higher range test will be negative also, and need not be performed.
- The same filtered extract can be used for both protocols if it is tightly capped between assays. The maximum time between assays cannot exceed one-half hour.
- Store the reagents at 4 °C when they are not in use. Allow the reagents to reach room temperature before using them in an analysis. Actual testing may be done at temperatures ranging from 1 °C to 38 °C.
- The Soil Extractant contains methyl alcohol which is poisonous and flammable. Before using this and other reagents, read the Material Safety Data Sheet (MSDS) for proper use of protective equipment and other safety information.

Note: *Hach Company recommends wearing protective nitrile gloves for this procedure.*

* This test is semi-quantitative. Results are expressed as greater or less than the threshold value used.

Soil Extraction Procedure

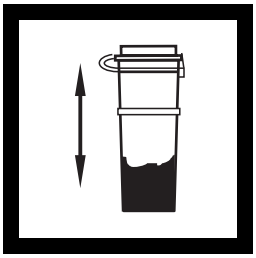
1. Weigh out 5 g of soil in the plastic weighing boat.



2. Carefully pour the soil into an extraction vial.

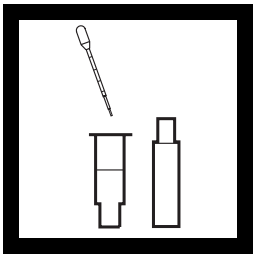
3. Use the 5-gram scoop to add one scoop of sodium sulfate to the extraction vial

4. Use the graduated cylinder to transfer 10 mL of Soil Extractant into the extraction vial.



5. Cap the extraction vial tightly and shake vigorously for one minute.

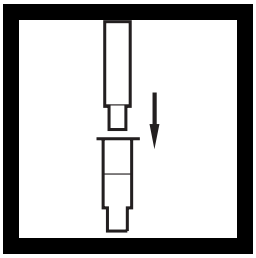
6. Allow to settle for at least one minute. Carefully open the extraction vial.



7. Using the disposable bulb pipet, withdraw 1.0 –1.5 mL from the liquid layer at the top of the extraction vial.

Transfer it into the filtration barrel (the bottom part of the filtering assembly into which the plunger inserts).

Note: *Do not use more than 1.5 mL. The bulb is marked in 0.25–mL increments.*

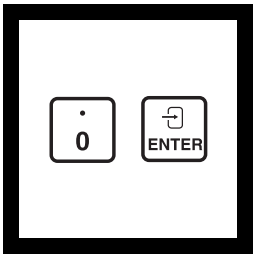


8. Insert the filtration plunger into the filtration barrel. Press firmly on the plunger until the sample extract is forced upward into the center of the plunger.

Use the resultant filtrate for the immunoassay in the Immunoassay Procedure for Soil Extracts

Note: *It may be necessary to place the filtration assembly on a table and press down on the plunger.*

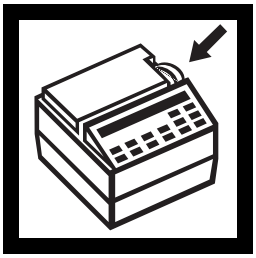
Immunoassay Procedure for Soil Extracts



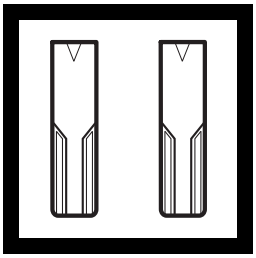
1. Enter the stored program for absorbance.

Press: **0 ENTER**

Note: *The Pour-Thru Cell cannot be used.*

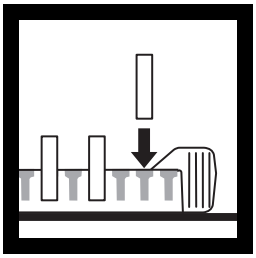


2. Dial the wavelength to 450 nm.

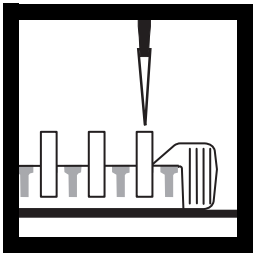


3. Label an Antibody Cuvette for each calibrator and each sample to be tested.

Note: *As many as 10 cuvettes may be tested at one time and may comprise any combination of samples and calibrators.*



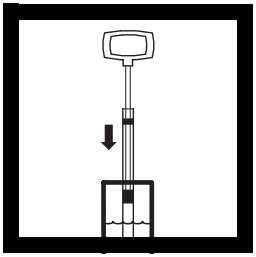
4. Place the cuvettes into the rack snugly.



5. Pipet 0.5 mL of Diluent Solution into each cuvette.

Note: *The same pipette tip can be used repeatedly for this step.*

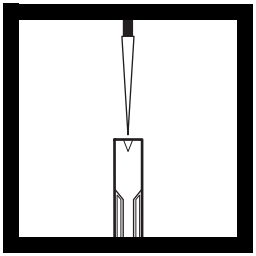
Note: *Have the necessary apparatus at hand for the next four steps as they must be done without delay.*



6. Use a Wiretrol® pipet to transfer the appropriate volume of calibrator or sample extract into each cuvette.

Note: *When testing at the 1 ppm and/or 5 ppm levels, use 50 µL of calibrator and sample. When testing at the 10 ppm and/or 50 ppm levels, use 10 µL of calibrator and sample.*

Note: *Use a separate capillary tube for each solution.*

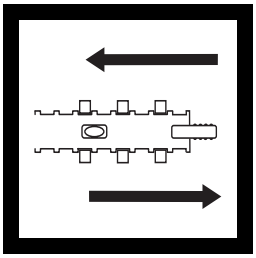


7. Immediately pipet 0.5 mL of PCB Enzyme Conjugate into each calibrator and sample cuvette.

Note: *The same pipette tip can be used repeatedly for this step.*

8. Press **SHIFT** then **7**. Enter 10 minutes and press **ENTER**.

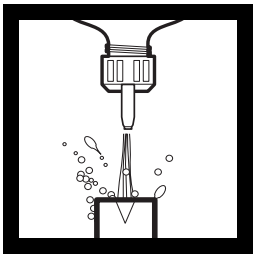
A 10-minute reaction time will begin. Proceed immediately to the next step.



9. Mix the contents of the cuvettes for 30 seconds using the technique described in *Using the 1-cm MicroCuvette Rack*.

10. After 5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique.

11. At the end of the 10-minute period, discard the contents of all the cuvettes into an appropriate waste container.

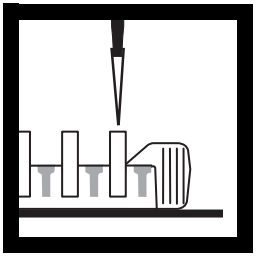


12. Wash each cuvette forcefully and thoroughly four times with deionized water. Empty the rinse water into the waste container.

Note: *Ensure most of the water is drained from the cuvettes by turning the cuvettes upside down and tapping them lightly on a paper towel*

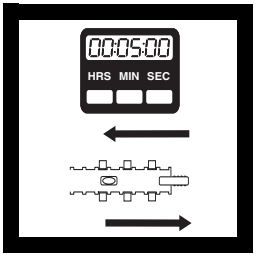
Color Development

Note: *Timing is critical; follow instructions carefully.*



13. With the cuvettes still held snugly in the rack, pipet 0.5 mL of Color Developing Solution into each Antibody Cuvette.

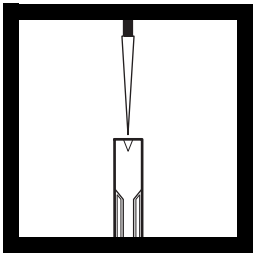
Note: *Use a new pipette tip for each cuvette.*



14. Press **SHIFT**, then **7**. Enter 5 minutes. Press **ENTER**.

A 5-minute reaction period will begin. Mix following the instructions in *Using the 1-cm MicroCuvette Rack*.

15. After 2.5 minutes, mix the contents of the rack a second time for a period of 30 seconds using the same technique.



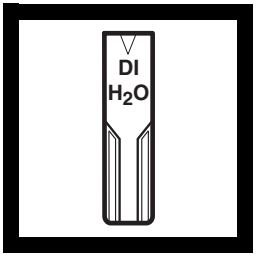
16. At the end of the 5-minute reaction period, pipette 0.5 mL of Stop Solution into each cuvette in the same order as the Color Developing Solution was added in step 12.

Slide the rack for 20 seconds using the technique described in *Using the 1-cm MicroCuvette Rack*.

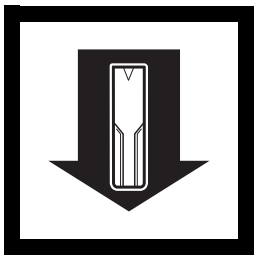
Note: *Blue solutions will turn yellow with the addition of the Stop Solution.*

Note: *The same pipette tip can be used repeatedly for this step.*

Measuring the Color

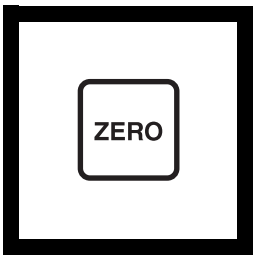


17. Label and fill a Zeroing Cuvette with deionized water. Wipe the outside of all the cuvettes with a tissue to remove water, smudges, and fingerprints.



18. Place the filled Zeroing cuvette into the cell holder.

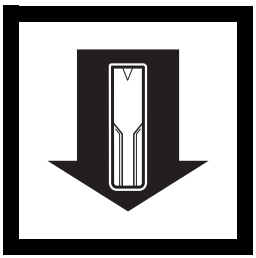
Orient the arrow to point towards the left side of the instrument for all cuvettes.



19. Press **ZERO**.

The display will show:

0.000 Abs



20. Place the prepared sample into the cell holder. Read the results.

The display will give an absorbance reading. Record the results for each calibrator and sample.

Note: *See the Instrument Manual for more information on taking a reading.*

21. Repeat step 19 for all remaining calibrators and samples.

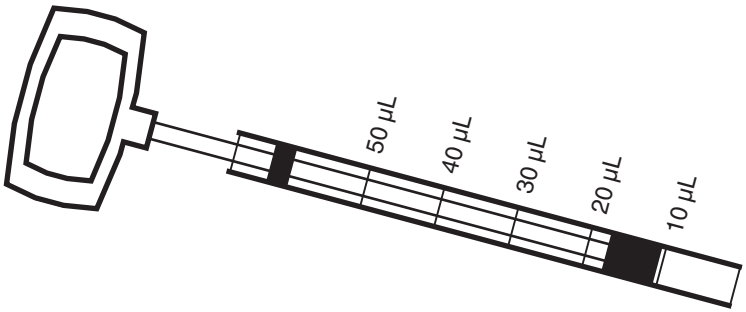
See Interpreting and Reporting Results for help with interpretation of results.

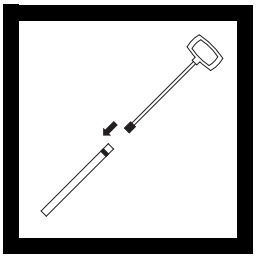
Using the Wiretrol® Pipet

The Wiretrol Pipet can accurately measure small quantities of liquids. It consists of two parts: a Teflon®-tipped plunger and a calibrated capillary tube. Use Figure 1 to determine the quantity measured at each line on the capillary tube.

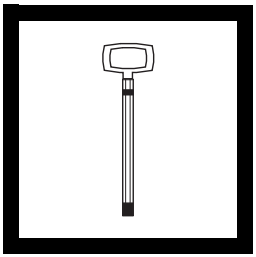
The plunger can be re-used; the capillary tubes must be discarded after one use.

Figure 1Wiretrol Pipet

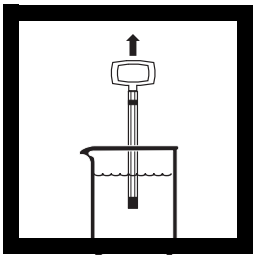




1. Wet the orange Teflon® tip of the Wiretrol plunger in the sample and carefully insert it into the end of the capillary tube with the colored band on it.

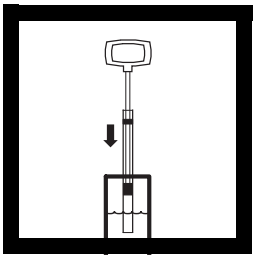


2. Push the tip to the other end of the capillary tube until it barely extends beyond the end of the capillary tube.



3. Submerge the capillary tube below the surface of the liquid to be pipetted. Slowly and smoothly draw the Wiretrol plunger up until the bottom of the plunger tips reaches the appropriate volume line.

Note: *Touch the end of the tube to the side of the vessel to release drops on the capillary tube tip.*

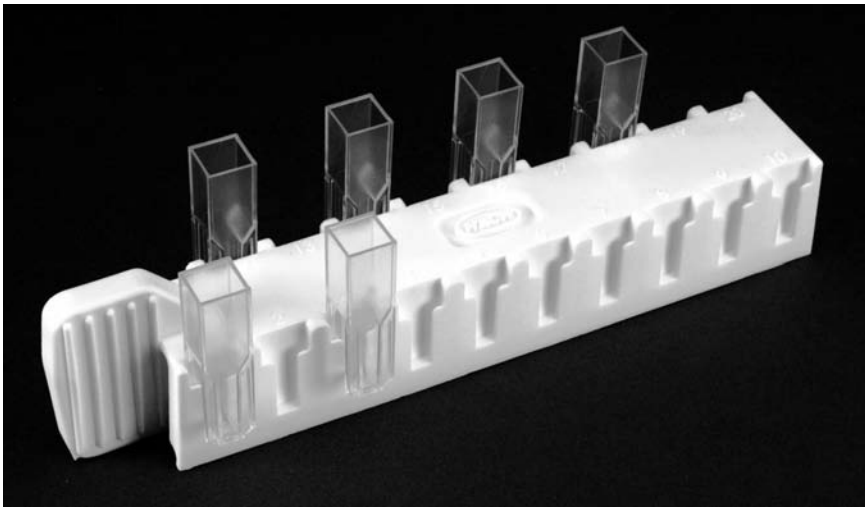


4. To discharge the pipet, place the tip of the capillary tube below the surface of the solution and push the Wiretrol plunger down in one smooth motion. Change capillary tubes for each calibrator and sample.

Using the 1-cm MicroCuvette Rack

This rack (see Figure 2) has been designed specifically to aid in achieving precise and accurate results when using the immunoassay technique to analyze several samples at the same time.

Figure 2The 1-cm MicroCuvette Rack



Loading the Rack —The cuvette rack is designed so that it may be inverted with the cuvettes in place. Identify each cuvette with a sample or calibrator number and place all the cuvettes in the rack before beginning the procedure. Fit the cuvettes snugly into the rack, but do not force them or they may be difficult to remove and their contents may spill. The cuvettes should remain in place when the rack is inverted and tapped lightly.

Mixing —Set the rack on a hard, flat surface that is at least twice the length of the rack. Hold the rack by one end and vigorously slide it back and forth along its long axis for 30 seconds. The rack should move through a distance equal to its own length in each direction.

Interpreting and Reporting Results

There is an inverse relationship between the concentration of PCB and the reading. In other words, the higher the reading, the lower the concentration of PCB.

If the sample reading is…	the sample PCB Concentration is…
…less than calibrator reading	…greater than the calibrator concentration
…greater than calibrator reading	…less than the calibrator concentration

Example

Readings:

1 ppm PCB Calibrator: **0.775 ABS**

5 ppm PCB Calibrator: **0.430 ABS**

Sample #1: **0.200 ABS**

Sample #2: **0.600 ABS**

Sample #3: **0.900 ABS**

Interpretation

Interpretation for a soil sample:

Sample #1 —Sample reading is less than the readings for both calibrators. Therefore the sample concentration of PCB is greater than both 1 ppm and 5 ppm as Aroclor 1248.

Sample #2 —Sample reading is between the readings for the 1 ppm and 5 ppm PCB calibrators. Therefore the sample concentration of PCB is between 1 ppm and 5 ppm as Aroclor 1248.

Sample #3 —Sample reading is greater than the readings for both calibrators. Therefore the sample concentration of PCB is less than both 5 ppm and 1 ppm as Aroclor 1248.

Storing and Handling Reagents

- Wear protective gloves and eyewear.
- When storing reagent sets for extended periods of time, keep them out of direct sunlight. Store reagents at a temperature of 4 °C when not in use.
- Keep the foil pouch containing the PCB Antibody Cuvettes sealed when not in use.
- If Stop Solution comes in contact with eyes, wash thoroughly for 15 minutes with cold water and seek immediate medical help.

Sensitivity

The PCB immunoassay cannot differentiate between the various Aroclors, but it detects their presence in differing degrees.

Table 1 Various PCBs in Soil

Compound	Concentration (ppm) to give a positive result at			
	1 ppm	5 ppm	10 ppm	50 ppm
1248	1	5	10	50
1016	2	9	20	67
1242	1.2	6	14	50
1254	1.4	4.6	11	28
1260	1.1	4.9	11	38

The following compounds are not detectable at 1000 ppm.

Biphenyl	2,4,6-trichlorophenyl	1,3-dichlorobenzene
2,4-dicholorophenyl	pentachlorophenol	1,4-dichlorobenzene
2,4,5-trichlorphenyl	1,2-dichlorobenzene	1,2,4-trichlorobenzene

Sample Collection and Storage

Analyze the samples as soon as possible after collection. If the samples must be stored, collect them in glass or Teflon® containers that have been washed with soap and water and rinsed with methanol. The container should be capped with a Teflon-lined cap. If a Teflon cap is not available, aluminum foil rinsed in methanol may be used as a substitute cap liner.

Summary of Method

Hach immunoassay tests use antigen/antibody reactions to test for specific organic compounds in water and soil. Antibodies specific for PCB are attached to the walls of plastic cuvettes. They selectively bind and remove PCB from complex sample matrices. A prepared sample and a reagent containing enzyme-conjugate molecules (analyte molecules attached to molecules of an enzyme) are added to the Antibody Cuvettes. During incubation, enzyme-conjugate molecules and PCB compete for binding sites on the antibodies. Samples with higher levels of analyte will have more antibody sites occupied by PCB and fewer antibody sites occupied by the enzyme-conjugate molecules.

After incubation, the sample and unbound enzyme conjugate are washed from the cuvette and a color-development reagent is added. The enzyme in the conjugate catalyzes the development of color. Therefore, there is an

inverse relationship between color intensity and the amount of PCB in the sample. The resulting color is then compared with a calibrator to determine whether the PCB concentration in the sample is greater or less than the threshold levels. The PCB concentration is inversely proportional to the color development: the lighter the color, the higher the PCB concentration.

Required Reagents

Description	Unit	Cat. No.
Reagent Set, PCB *	20 cuvettes	27735-00
Deionized water	500 mL	272-48

Required Apparatus

Adapter, 1-cm MicroCell	each	49349-00
Caps, flip spout	2/pkg	25818-02
Marker, laboratory	each	20920-00
TenSette®, Pipet, 0.1–1.0 mL	each	19000-01
Tips, for TenSette®, Pipet19000-01	1000/pkg	21856-28
Rack, for 1-cm Micro Cuvettes	each	48799-00
Wipes, disposable	box	20970-00

For Soil Extraction only:

Soil Scoop, 5 g, 4.25 cc	each	26572-05
Soil Extraction Refill Kit	each	27752-00
Includes:		
Dropper, LDPE, 0.5 and 1.0-mL,	20/pkg	21247-20
Filter and Barrel Assembly	20/pkg	25676-20
Sodium Sulfate, anhydrous	250 g	7099-29
Soil Extractant Solution	200 mL	25677-29
Soil Sample Container	20/pkg	25929-20
Weighing Boat, 8.9 cm square	20/pkg	21790-20

* Immunoassay components are manufactured for Hach Company by Beacon Analytical Systems, Inc.

METHOD 6200

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the in situ and intrusive analysis of the 26 analytes listed in Table 1 for soil and sediment samples. Some common elements are not listed in Table 1 because they are considered "light" elements that cannot be detected by field portable x-ray fluorescence (FPXRF). They are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Most of the analytes listed in Table 1 are of environmental concern, while a few others have interference effects or change the elemental composition of the matrix, affecting quantitation of the analytes of interest. Generally elements of atomic number 16 or greater can be detected and quantitated by FPXRF.

1.2 Detection limits depend on several factors, the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, count times used to irradiate the sample, physical matrix effects, chemical matrix effects, and interelement spectral interferences. General instrument detection limits for analytes of interest in environmental applications are shown in Table 1. These detection limits apply to a clean matrix of quartz sand (silicon dioxide) free of interelement spectral interferences using long (600-second) count times. These detection limits are given for guidance only and will vary depending on the sample matrix, which instrument is used, and operating conditions. A discussion of field performance-based detection limits is presented in Section 13.4 of this method. The clean matrix and field performance-based detection limits should be used for general planning purposes, and a third detection limit discussed, based on the standard deviation around single measurements, should be used in assessing data quality. This detection limit is discussed in Sections 9.7 and 11.3.

1.3 Use of this method is restricted to personnel either trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. This method is a screening method to be used with confirmatory analysis using EPA-approved methods. This method's main strength is as a rapid field screening procedure. The method detection limits (MDL) of FPXRF are above the toxicity characteristic regulatory level for most RCRA analytes. If the precision, accuracy, and detection limits of FPXRF meet the data quality objectives (DQOs) of your project, then XRF is a fast, powerful, cost effective technology for site characterization.

2.0 SUMMARY OF METHOD

2.1 The FPXRF technologies described in this method use sealed radioisotope sources to irradiate samples with x-rays. X-ray tubes are used to irradiate samples in the laboratory and are beginning to be incorporated into field portable instruments. When a sample is irradiated with x-rays, the source x-rays may undergo either scattering or absorption by sample atoms. This later process is known as the photoelectric effect. When an atom absorbs the source x-rays, the incident radiation dislodges electrons from the innermost shells of the atom, creating vacancies. The electron vacancies are filled by electrons cascading in from outer electron shells. Electrons in outer shells have higher energy states than inner shell electrons, and the outer shell electrons give off energy as they cascade down into the inner shell vacancies. This rearrangement of electrons

results in emission of x-rays characteristic of the given atom. The emission of x-rays, in this manner, is termed x-ray fluorescence.

Three electron shells are generally involved in emission of x-rays during FPXRF analysis of environmental samples: the K, L, and M shells. A typical emission pattern, also called an emission spectrum, for a given metal has multiple intensity peaks generated from the emission of K, L, or M shell electrons. The most commonly measured x-ray emissions are from the K and L shells; only metals with an atomic number greater than 57 have measurable M shell emissions.

Each characteristic x-ray line is defined with the letter K, L, or M, which signifies which shell had the original vacancy and by a subscript alpha (α) or beta (β), which indicates the higher shell from which electrons fell to fill the vacancy and produce the x-ray. For example, a K_α line is produced by a vacancy in the K shell filled by an L shell electron, whereas a K_β line is produced by a vacancy in the K shell filled by an M shell electron. The K_α transition is on average 6 to 7 times more probable than the K_β transition; therefore, the K_α line is approximately 7 times more intense than the K_β line for a given element, making the K_α line the choice for quantitation purposes.

The K lines for a given element are the most energetic lines and are the preferred lines for analysis. For a given atom, the x-rays emitted from L transitions are always less energetic than those emitted from K transitions. Unlike the K lines, the main L emission lines (L_α and L_β) for an element are of nearly equal intensity. The choice of one or the other depends on what interfering element lines might be present. The L emission lines are useful for analyses involving elements of atomic number (Z) 58 (cerium) through 92 (uranium).

An x-ray source can excite characteristic x-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element, that is, the K absorption edge, L absorption edge, or M absorption edge energy. The absorption edge energy is somewhat greater than the corresponding line energy. Actually, the K absorption edge energy is approximately the sum of the K, L, and M line energies of the particular element, and the L absorption edge energy is approximately the sum of the L and M line energies. FPXRF is more sensitive to an element with an absorption edge energy close to but less than the excitation energy of the source. For example, when using a cadmium-109 source, which has an excitation energy of 22.1 kiloelectron volts (keV), FPXRF would exhibit better sensitivity for zirconium which has a K line energy of 15.7 keV than to chromium, which has a K line energy of 5.41 keV.

2.2 Under this method, inorganic analytes of interest are identified and quantitated using a field portable energy-dispersive x-ray fluorescence spectrometer. Radiation from one or more radioisotope sources or an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. Up to three sources may be used to irradiate a sample. Each source emits a specific set of primary x-rays that excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the source is selected according to its excitation efficiency for the element of interest.

For measurement, the sample is positioned in front of the probe window. This can be done in two manners using FPXRF instruments: in situ or intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. The sample cup is then placed on top of the window inside a protective cover for analysis.

Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PC).

The measurement time of each source is user-selectable. Shorter source measurement times (30 seconds) are generally used for initial screening and hot spot delineation, and longer measurement times (up to 300 seconds) are typically used to meet higher precision and accuracy requirements.

FPXRF instruments can be calibrated using the following methods: internally using fundamental parameters determined by the manufacturer, empirically based on site-specific calibration standards (SSCS), or based on Compton peak ratios. The Compton peak is produced by backscattering of the source radiation. Some FPXRF instruments can be calibrated using multiple methods.

3.0 DEFINITIONS

3.1 FPXRF: Field portable x-ray fluorescence.

3.2 MCA: Multichannel analyzer for measuring pulse amplitude.

3.3 SSCS: Site specific calibration standard.

3.4 FP: Fundamental parameter.

3.5 ROI: Region of interest.

3.6 SRM: Standard reference material. A standard containing certified amounts of metals in soil or sediment.

3.7 eV: Electron Volt. A unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.

3.8 Refer to Chapter One and Chapter Three for additional definitions.

4.0 INTERFERENCES

4.1 The total method error for FPXRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in FPXRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

4.2 Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup. One way to reduce such error is to grind and sieve all soil samples to a uniform particle size thus reducing sample-to-sample particle size variability. Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples.

4.3 Moisture content may affect the accuracy of analysis of soil and sediment sample analyses. When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water. This error can be minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

4.4 Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

4.5 Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of iron. The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using SSCS, which contain all the elements present on site that can interfere with one another.

4.6 When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum. The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K_β line of element Z-1 with the K_α line of element Z. This is called the K_α/K_β interference. Because the K_α:K_β intensity ratio for a given element usually is about 7:1, the interfering element, Z-1, must be present at large concentrations to cause a problem. Two examples of this type of spectral interference involve the presence of large concentrations of vanadium (V) when attempting to measure Cr or the presence of large concentrations of Fe when attempting to measure cobalt (Co). The V K_α and K_β energies are 4.95

and 5.43 keV, respectively, and the Cr K_α energy is 5.41 keV. The Fe K_α and K_β energies are 6.40 and 7.06 keV, respectively, and the Co K_α energy is 6.92 keV. The difference between the V K_β and Cr K_α energies is 20 eV, and the difference between the Fe K_β and the Co K_α energies is 140 eV. The resolution of the highest-resolution detectors in FPXRF instruments is 170 eV. Therefore, large amounts of V and Fe will interfere with quantitation of Cr or Co, respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

4.7 Other interferences can arise from K/L, K/M, and L/M line overlaps, although these overlaps are less common. Examples of such overlap involve arsenic (As) K_α/lead (Pb) L_α and sulfur (S) K_α/Pb M_α. In the As/Pb case, Pb can be measured from the Pb L_β line, and As can be measured from either the As K_α or the As $K_ß$ line; in this way the interference can be corrected. If the As K_β line is used, sensitivity will be decreased by a factor of two to five times because it is a less intense line than the As K_α line. If the As K_α line is used in the presence of Pb, mathematical corrections within the instrument software can be used to subtract out the Pb interference. However, because of the limits of mathematical corrections, As concentrations cannot be efficiently calculated for samples with Pb:As ratios of 10:1 or more. This high ratio of Pb to As may result in no As being reported regardless of the actual concentration present.

No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. The operator's decision will be based on action levels for metals in soil established for the site, matrix effects, capabilities of the instrument, data quality objectives, and the ratio of lead to arsenic known to be present at the site. If a site is encountered that contains lead at concentrations greater than ten times the concentration of arsenic it is advisable that all critical soil samples be sent off site for confirmatory analysis by an EPA-approved method.

4.8 If SSCS are used to calibrate an FPXRF instrument, the samples collected must be representative of the site under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentrations of the contaminants of concern at a given time and location. Analytical results for representative samples reflect variations in the presence and concentration ranges of contaminants throughout a site. Variables affecting sample representativeness include differences in soil type, contaminant concentration variability, sample collection and preparation variability, and analytical variability, all of which should be minimized as much as possible.

4.9 Soil physical and chemical effects may be corrected using SSCS that have been analyzed by inductively coupled plasma (ICP) or atomic absorption (AA) methods. However, a major source of error can be introduced if these samples are not representative of the site or if the analytical error is large. Another concern is the type of digestion procedure used to prepare the soil samples for the reference analysis. Analytical results for the confirmatory method will vary depending on whether a partial digestion procedure, such as SW-846 Method 3050, or a total digestion procedure, such as Method 3052 is used. It is known that depending on the nature of the soil or sediment, Method 3050 will achieve differing extraction efficiencies for different analytes of interest. The confirmatory method should meet the project data quality objectives.

XRF measures the total concentration of an element; therefore, to achieve the greatest comparability of this method with the reference method (reduced bias), a total digestion procedure should be used for sample preparation. However, in the study used to generate the performance data for this method, the confirmatory method used was Method 3050, and the FPXRF data

compared very well with regression correlation coefficients (r^2 often exceeding 0.95, except for barium and chromium. See Table 9 in Section 17.0). The critical factor is that the digestion procedure and analytical reference method used should meet the data quality objectives (DQOs) of the project and match the method used for confirmation analysis.

4.10 Ambient temperature changes can affect the gain of the amplifiers producing instrument drift. Gain or drift is primarily a function of the electronics (amplifier or preamplifier) and not the detector as most instrument detectors are cooled to a constant temperature. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration. The performance of an energy calibration check to assess drift is a quality control measure discussed in Section 9.2.

If the operator is instructed by the manufacturer to manually conduct a gain check because of increasing or decreasing ambient temperature, it is standard to perform a gain check after every 10 to 20 sample measurements or once an hour whichever is more frequent. It is also suggested that a gain check be performed if the temperature fluctuates more than 10 to 20°F. The operator should follow the manufacturer's recommendations for gain check frequency.

5.0 SAFETY

5.1 Proper training for the safe operation of the instrument and radiation training should be completed by the analyst prior to analysis. Radiation safety for each specific instrument can be found in the operators manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals. A copy of the radioactive material licenses and leak tests should be present with the instrument at all times and available to local and national authorities upon request. X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.2 Radiation monitoring equipment should be used with the handling of the instrument. The operator and the surrounding environment should be monitored continually for analyst exposure to radiation. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. The TLDs should be worn in the area of most frequent exposure. The maximum permissible whole-body dose from occupational exposure is 5

Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding.

5.3 Refer to Chapter Three for guidance on some proper safety protocols.

6.0 EQUIPMENT AND SUPPLIES

6.1 FPXRF Spectrometer: An FPXRF spectrometer consists of four major components: (1) a source that provides x-rays; (2) a sample presentation device; (3) a detector that converts x-ray-generated photons emitted from the sample into measurable electronic signals; and (4) a data processing unit that contains an emission or fluorescence energy analyzer, such as an MCA, that processes the signals into an x-ray energy spectrum from which elemental concentrations in the sample may be calculated, and a data display and storage system. These components and additional, optional items, are discussed below.

6.1.1 Excitation Sources: Most FPXRF instruments use sealed radioisotope sources to produce x-rays in order to irradiate samples. The FPXRF instrument may contain between one and three radioisotope sources. Common radioisotope sources used for analysis for metals in soils are iron (Fe)-55, cadmium (Cd)-109, americium (Am)-241, and curium (Cm)-244. These sources may be contained in a probe along with a window and the detector; the probe is connected to a data reduction and handling system by means of a flexible cable. Alternatively, the sources, window, and detector may be included in the same unit as the data reduction and handling system.

The relative strength of the radioisotope sources is measured in units of millicuries (mCi). All other components of the FPXRF system being equal, the stronger the source, the greater the sensitivity and precision of a given instrument. Radioisotope sources undergo constant decay. In fact, it is this decay process that emits the primary x-rays used to excite samples for FPXRF analysis. The decay of radioisotopes is measured in "half-lives." The half-life of a radioisotope is defined as the length of time required to reduce the radioisotopes strength or activity by half. Developers of FPXRF technologies recommend source replacement at regular intervals based on the source's half-life. The characteristic x-rays emitted from each of the different sources have energies capable of exciting a certain range of analytes in a sample. Table 2 summarizes the characteristics of four common radioisotope sources.

X-ray tubes have higher radiation output, no intrinsic lifetime limit, produce constant output over their lifetime, and do not have the disposal problems of radioactive sources but are just now appearing in FPXRF instruments An electrically-excited x-ray tube operates by bombarding an anode with electrons accelerated by a high voltage. The electrons gain an energy in electron volts equal to the accelerating voltage and can excite atomic transitions in the anode, which then produces characteristic x-rays. These characteristic x-rays are emitted through a window which contains the vacuum required for the electron acceleration. An important difference between x-ray tubes and radioactive sources is that the electrons which bombard the anode also produce a continuum of x-rays across a broad range of energies in addition to the characteristic x-rays. This continuum is weak compared to the characteristic x-rays but can provide substantial excitation since it covers a broad energy range. It has the undesired property of producing background in the spectrum near the analyte x-ray lines when it is scattered by the sample. For this reason a filter is often used between the x-ray tube and the sample to suppress the continuum radiation while passing the characteristic

x-rays from the anode. This filter is sometimes incorporated into the window of the x-ray tube. The choice of accelerating voltage is governed by the anode material, since the electrons must have sufficient energy to excite the anode, which requires a voltage greater than the absorption edge of the anode material. The anode is most efficiently excited by voltages 2 to 2.5 times the edge energy (most x-rays per unit power to the tube), although voltages as low as 1.5 times the absorption edge energy will work. The characteristic x-rays emitted by the anode are capable of exciting a range of elements in the sample just as with a radioactive source. Table 3 gives the recommended operating voltages and the sample elements excited for some common anodes.

6.1.2 Sample Presentation Device: FPXRF instruments can be operated in two modes: in situ and intrusive. If operated in the in situ mode, the probe window is placed in direct contact with the soil surface to be analyzed. When an FPXRF instrument is operated in the intrusive mode, a soil or sediment sample must be collected, prepared, and placed in a sample cup. For most FPXRF instruments operated in the intrusive mode, the probe is rotated so that the window faces upward. A protective sample cover is placed over the window, and the sample cup is placed on top of the window inside the protective sample cover for analysis.

6.1.3 Detectors: The detectors in the FPXRF instruments can be either solid-state detectors or gas-filled, proportional counter detectors. Common solid-state detectors include mercuric iodide (HgI_2), silicon pin diode and lithium-drifted silicon Si(Li). The HgI_2 detector is operated at a moderately subambient temperature controlled by a low power thermoelectric cooler. The silicon pin diode detector also is cooled via the thermoelectric Peltier effect. The Si(Li) detector must be cooled to at least -90 °C either with liquid nitrogen or by thermoelectric cooling via the Peltier effect. Instruments with a Si(Li) detector have an internal liquid nitrogen dewar with a capacity of 0.5 to 1.0 liter. Proportional counter detectors are rugged and lightweight, which are important features of a field portable detector. However, the resolution of a proportional counter detector is not as good as that of a solid-state detector. The energy resolution of a detector for characteristic x-rays is usually expressed in terms of full width at half-maximum (FWHM) height of the manganese K_α peak at 5.89 keV. The typical resolutions of the above mentioned detectors are as follows: HgI_2-270 eV; silicon pin diode-250 eV; Si(Li)–170 eV; and gas-filled, proportional counter-750 eV.

During operation of a solid-state detector, an x-ray photon strikes a biased, solid-state crystal and loses energy in the crystal by producing electron-hole pairs. The electric charge produced is collected and provides a current pulse that is directly proportional to the energy of the x-ray photon absorbed by the crystal of the detector. A gas-filled, proportional counter detector is an ionization chamber filled with a mixture of noble and other gases. An x-ray photon entering the chamber ionizes the gas atoms. The electric charge produced is collected and provides an electric signal that is directly proportional to the energy of the x-ray photon absorbed by the gas in the detector.

6.1.4 Data Processing Units: The key component in the data processing unit of an FPXRF instrument is the MCA. The MCA receives pulses from the detector and sorts them by their amptitudes (energy level). The MCA counts pulses per second to determine the height of the peak in a spectrum, which is indicative of the target analyte's concentration. The spectrum of element peaks are built on the MCA. The MCAs in FPXRF instruments have from 256 to 2,048 channels. The concentrations of target analytes are usually shown in parts per million on a liquid crystal display (LCD) in the instrument. FPXRF instruments can store both spectra and from 100 to 500 sets of numerical analytical results. Most FPXRF

instruments are menu-driven from software built into the units or from PCs. Once the data–storage memory of an FPXRF unit is full, data can be downloaded by means of an RS-232 port and cable to a PC.

6.2 Spare battery chargers.

6.3 Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

6.4 X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

6.5 Mortar and pestle: glass, agate, or aluminum oxide; for grinding soil and sediment samples.

6.6 Containers: glass or plastic to store samples.

6.7 Sieves: 60-mesh (0.25 mm), stainless-steel, Nylon, or equivalent for preparing soil and sediment samples.

6.8 Trowels: for smoothing soil surfaces and collecting soil samples.

6.9 Plastic bags: used for collection and homogenization of soil samples.

6.10 Drying oven: standard convection or toaster oven, for soil and sediment samples that require drying.

7.0 REAGENTS AND STANDARDS

7.1 Pure Element Standards: Each pure, single-element standard is intended to produce strong characteristic x-ray peaks of the element of interest only. Other elements present must not contribute to the fluorescence spectrum. A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if required for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

7.2 Site-specific Calibration Standards: Instruments that employ fundamental parameters (FP) or similar mathematical models in minimizing matrix effects may not require SSCS. If the FP calibration model is to be optimized or if empirical calibration is necessary, then SSCSs must be collected, prepared, and analyzed.

7.2.1 The SSCS must be representative of the matrix to be analyzed by FPXRF. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site. A sample size of 4 to 8 ounces is recommended, and standard glass sampling jars should be used.

7.2.2 Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C. If mercury is to be analyzed, a separate sample portion must remain undried, as heating may volatilize the mercury. When the sample is dry, all large, organic debris and

nonrepresentative material, such as twigs, leaves, roots, insects, asphalt, and rock should be removed. The sample should be ground with a mortar and pestle and passed through a 60-mesh sieve. Only the coarse rock fraction should remain on the screen.

7.2.3 The sample should be homogenized by using a riffle splitter or by placing 150 to 200 grams of the dried, sieved sample on a piece of kraft or butcher paper about 1.5 by 1.5 feet in size. Each corner of the paper should be lifted alternately, rolling the soil over on itself and toward the opposite corner. The soil should be rolled on itself 20 times. Approximately 5 grams of the sample should then be removed and placed in a sample cup for FPXRF analysis. The rest of the prepared sample should be sent off site for ICP or AA analysis. The method use for confirmatory analysis should meet the data quality objectives of the project.

7.3 Blank Samples: The blank samples should be from a "clean" quartz or silicon dioxide matrix that is free of any analytes at concentrations above the method detection limits. These samples are used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

7.4 Standard Reference Materials: Standard reference materials (SRM) are standards containing certified amounts of metals in soil or sediment. These standards are used for accuracy and performance checks of FPXRF analyses. SRMs can be obtained from the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample handling and preservation procedures used in FPXRF analyses should follow the guidelines in Chapter Three, Inorganic Analytes.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for additional guidance on quality assurance protocols. All field data sheets and quality control data should be maintained for reference or inspection.

9.2 Energy Calibration Check: To determine whether an FPXRF instrument is operating within resolution and stability tolerances, an energy calibration check should be run. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. As discussed in Section 4.10, this check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

The energy calibration check should be run at a frequency consistent with manufacturers recommendations. Generally, this would be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead is often used for the energy calibration check. A manufacturer-recommended count time per source should be used for the check.

9.2.1 The instrument manufacturer's manual specifies the channel or kiloelectron volt level at which a pure element peak should appear and the expected intensity of the peak.

The intensity and channel number of the pure element as measured using the radioactive source should be checked and compared to the manufacturer's recommendation. If the energy calibration check does not meet the manufacturer's criteria, then the pure element sample should be repositioned and reanalyzed. If the criteria are still not met, then an energy calibration should be performed as described in the manufacturer's manual. With some FPXRF instruments, once a spectrum is acquired from the energy calibration check, the peak can be optimized and realigned to the manufacturer's specifications using their software.

9.3 Blank Samples: Two types of blank samples should be analyzed for FPXRF analysis: instrument blanks and method blanks. An instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

9.3.1 The instrument blank can be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate. This instrument blank should be analyzed on each working day before and after analyses are conducted and once per every twenty samples. An instrument blank should also be analyzed whenever contamination is suspected by the analyst. The frequency of analysis will vary with the data quality objectives of the project. A manufacturer-recommended count time per source should be used for the blank analysis. No element concentrations above the method detection limits should be found in the instrument blank. If concentrations exceed these limits, then the probe window and the check sample should be checked for contamination. If contamination is not a problem, then the instrument must be "zeroed" by following the manufacturer's instructions.

9.3.2 A method blank is used to monitor for laboratory-induced contaminants or interferences. The method blank can be "clean" silica sand or lithium carbonate that undergoes the same preparation procedure as the samples. A method blank must be analyzed at least daily. The frequency of analysis will depend on the data quality objectives of the project. To be acceptable, a method blank must not contain any analyte at a concentration above its method detection limit. If an analyte's concentration exceeds its method detection limit, the cause of the problem must be identified, and all samples analyzed with the method blank must be reanalyzed.

9.4 Calibration Verification Checks: A calibration verification check sample is used to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest. A check sample should be analyzed at the beginning of each working day, during active sample analyses, and at the end of each working day. The frequency of calibration checks during active analysis will depend on the data quality objectives of the project. The check sample should be a well characterized soil sample from the site that is representative of site samples in terms of particle size and degree of homogeneity and that contains contaminants at concentrations near the action levels. If a site-specific sample is not available, then an NIST or other SRM that contains the analytes of interest can be used to verify the accuracy of the instrument. The measured value for each target analyte should be within ±20 percent (%D) of the true value for the calibration verification check to be acceptable. If a measured value falls outside this range, then the check sample should be reanalyzed. If the value continues to fall outside the acceptance range, the instrument should be recalibrated, and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

9.5 Precision Measurements: The precision of the method is monitored by analyzing a sample with low, moderate, or high concentrations of target analytes. The frequency of precision measurements will depend on the data quality objectives for the data. A minimum of one precision sample should be run per day. Each precision sample should be analyzed 7 times in replicate. It

is recommended that precision measurements be obtained for samples with varying concentration ranges to assess the effect of concentration on method precision. Determining method precision for analytes at concentrations near the site action levels can be extremely important if the FPXRF results are to be used in an enforcement action; therefore, selection of at least one sample with target analyte concentrations at or near the site action levels or levels of concern is recommended. A precision sample is analyzed by the instrument for the same field analysis time as used for other project samples. The relative standard deviation (RSD) of the sample mean is used to assess method precision. For FPXRF data to be considered adequately precise, the RSD should not be greater than 20 percent with the exception of chromium. RSD values for chromium should not be greater than 30 percent.

The equation for calculating RSD is as follows:

$$RSD = (SD/\text{Mean Concentration}) \times 100$$

where:

RSD	=	Relative standard deviation for the precision measurement for the analyte
SD	=	Standard deviation of the concentration for the analyte
Mean Concentration	=	Mean concentration for the analyte

The precision or reproducibility of a measurement will improve with increasing count time, however, increasing the count time by a factor of 4 will provide only 2 times better precision, so there is a point of diminishing return. Increasing the count time also improves the detection limit, but decreases sample throughput.

9.6 Detection Limits: Results for replicate analyses of a low-concentration sample, SSCS, or SRM can be used to generate an average site-specific method detection and quantitation limits. In this case, the method detection limit is defined as 3 times the standard deviation of the results for the low-concentration samples and the method quantitation limit is defined as 10 times the standard deviation of the same results. Another means of determining method detection and quantitation limits involves use of counting statistics. In FPXRF analysis, the standard deviation from counting statistics is defined as $SD = (N)^{1/2}$, where SD is the standard deviation for a target analyte peak and N is the net counts for the peak of the analyte of interest (i.e., gross counts minus background under the peak). Three times this standard deviation would be the method detection limit and 10 times this standard deviation would be the method quantitation limit. If both of the above mentioned approaches are used to calculate method detection limits, the larger of the standard deviations should be used to provide the more conservative detection limits.

This SD based detection limit criteria must be used by the operator to evaluate each measurement for its useability. A measurement above the average calculated or manufacturer's detection limit, but smaller than three times its associated SD, should not be used as a quantitative measurement. Conversely, if the measurement is below the average calculated or manufacturer's detection limit, but greater than three times its associated SD. It should be coded as an estimated value.

9.7 Confirmatory Samples: The comparability of the FPXRF analysis is determined by submitting FPXRF-analyzed samples for analysis at a laboratory. The method of confirmatory analysis must meet the project and XRF measurement data quality objectives. The confirmatory samples must be splits of the well homogenized sample material. In some cases the prepared

sample cups can be submitted. A minimum of 1 sample for each 20 FPXRF-analyzed samples should be submitted for confirmatory analysis. This frequency will depend on data quality objectives. The confirmatory analyses can also be used to verify the quality of the FPXRF data. The confirmatory samples should be selected from the lower, middle, and upper range of concentrations measured by the FPXRF. They should also include samples with analyte concentrations at or near the site action levels. The results of the confirmatory analysis and FPXRF analyses should be evaluated with a least squares linear regression analysis. If the measured concentrations span more than one order of magnitude, the data should be log-transformed to standardize variance which is proportional to the magnitude of measurement. The correlation coefficient (r^2) for the results should be 0.7 or greater for the FPXRF data to be considered screening level data. If the r^2 is 0.9 or greater and inferential statistics indicate the FPXRF data and the confirmatory data are statistically equivalent at a 99 percent confidence level, the data could potentially meet definitive level data criteria.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Instrument Calibration: Instrument calibration procedures vary among FPXRF instruments. Users of this method should follow the calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, however, three types of calibration procedures exist for FPXRF instruments: FP calibration, empirical calibration, and the Compton peak ratio or normalization method. These three types of calibration are discussed below.

10.2 Fundamental Parameters Calibration: FP calibration procedures are extremely variable. An FP calibration provides the analyst with a "standardless" calibration. The advantages of FP calibrations over empirical calibrations include the following:

- No previously collected site-specific samples are required, although site-specific samples with confirmed and validated analytical results for all elements present could be used.

- Cost is reduced because fewer confirmatory laboratory results or calibration standards are required.

However, the analyst should be aware of the limitations imposed on FP calibration by particle size and matrix effects. These limitations can be minimized by adhering to the preparation procedure described in Section 7.2. The two FP calibration processes discussed below are based on an effective energy FP routine and a back scatter with FP (BFP) routine. Each FPXRF FP calibration process is based on a different iterative algorithmic method. The calibration procedure for each routine is explained in detail in the manufacturer's user manual for each FPXRF instrument; in addition, training courses are offered for each instrument.

10.2.1 Effective Energy FP Calibration: The effective energy FP calibration is performed by the manufacturer before an instrument is sent to the analyst. Although SSCS can be used, the calibration relies on pure element standards or SRMs such as those obtained from NIST for the FP calibration. The effective energy routine relies on the spectrometer response to pure elements and FP iterative algorithms to compensate for various matrix effects.

Alpha coefficients are calculated using a variation of the Sherman equation, which calculates theoretical intensities from the measurement of pure element samples. These coefficients indicate the quantitative effect of each matrix element on an analyte's measured

x-ray intensity. Next, the Lachance Traill algorithm is solved as a set of simultaneous equations based on the theoretical intensities. The alpha coefficients are then downloaded into the specific instrument.

The working effective energy FP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of sampling. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. A manufacturer-recommended count time per source should be used for the calibration check. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A percent difference (%D) is then calculated for each target analyte. The %D should be within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line or the y-intercept value for the analyte. The SRM or SSCS is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

The equation to calibrate %D is as follows:

$$\%D = ((C_s - C_k) / C_k) \times 100$$

where:

%D = Percent difference
C_k = Certified concentration of standard sample
C_s = Measured concentration of standard sample

10.2.2 BFP Calibration: BFP calibration relies on the ability of the liquid nitrogen-cooled, Si(Li) solid-state detector to separate the coherent (Compton) and incoherent (Rayleigh) backscatter peaks of primary radiation. These peak intensities are known to be a function of sample composition, and the ratio of the Compton to Rayleigh peak is a function of the mass absorption of the sample. The calibration procedure is explained in detail in the instrument manufacturer's manual. Following is a general description of the BFP calibration procedure.

The concentrations of all detected and quantified elements are entered into the computer software system. Certified element results for an NIST SRM or confirmed and validated results for an SSCS can be used. In addition, the concentrations of oxygen and silicon must be entered; these two concentrations are not found in standard metals analyses. The manufacturer provides silicon and oxygen concentrations for typical soil types. Pure element standards are then analyzed using a manufacturer-recommended count time per source. The results are used to calculate correction factors in order to adjust for spectrum overlap of elements.

The working BFP calibration curve must be verified before sample analysis begins on each working day, after every 20 samples are analyzed, and at the end of the analysis. This verification is performed by analyzing either an NIST SRM or an SSCS that is representative of the site-specific samples. This SRM or SSCS serves as a calibration check. The standard sample is analyzed using a manufacturer-recommended count time per source to check the

calibration curve. The analyst must then adjust the y-intercept and slope of the calibration curve to best fit the known concentrations of target analytes in the SRM or SSCS.

A %D is then calculated for each target analyte. The %D should fall within ±20 percent of the certified value for each analyte. If the %D falls outside this acceptance range, then the calibration curve should be adjusted by varying the slope of the line the y-intercept value for the analyte. The standard sample is reanalyzed until the %D falls within ±20 percent. The group of 20 samples analyzed before an out-of-control calibration check should be reanalyzed.

10.3 Empirical Calibration: An empirical calibration can be performed with SSCS, site-typical standards, or standards prepared from metal oxides. A discussion of SSCS is included in Section 7.2; if no previously characterized samples exist for a specific site, site-typical standards can be used. Site-typical standards may be selected from commercially available characterized soils or from SSCS prepared for another site. The site-typical standards should closely approximate the site's soil matrix with respect to particle size distribution, mineralogy, and contaminant analytes. If neither SSCS nor site-typical standards are available, it is possible to make gravimetric standards by adding metal oxides to a "clean" sand or silicon dioxide matrix that simulates soil. Metal oxides can be purchased from various chemical vendors. If standards are made on site, a balance capable of weighing items to at least two decimal places is required. Concentrated ICP or AA standard solutions can also be used to make standards. These solutions are available in concentrations of 10,000 parts per million, thus only small volumes have to be added to the soil.

An empirical calibration using SSCS involves analysis of SSCS by the FPXRF instrument and by a conventional analytical method such as ICP or AA. A total acid digestion procedure should be used by the laboratory for sample preparation. Generally, a minimum of 10 and a maximum of 30 well characterized SSCS, site-typical standards, or prepared metal oxide standards are required to perform an adequate empirical calibration. The number of required standards depends on the number of analytes of interest and interfering elements. Theoretically, an empirical calibration with SSCS should provide the most accurate data for a site because the calibration compensates for site-specific matrix effects.

The first step in an empirical calibration is to analyze the pure element standards for the elements of interest. This enables the instrument to set channel limits for each element for spectral deconvolution. Next the SSCS, site-typical standards, or prepared metal oxide standards are analyzed using a count time of 200 seconds per source or a count time recommended by the manufacturer. This will produce a spectrum and net intensity of each analyte in each standard. The analyte concentrations for each standard are then entered into the instrument software; these concentrations are those obtained from the laboratory, the certified results, or the gravimetrically determined concentrations of the prepared standards. This gives the instrument analyte values to regress against corresponding intensities during the modeling stage. The regression equation correlates the concentrations of an analyte with its net intensity.

The calibration equation is developed using a least squares fit regression analysis. After the regression terms to be used in the equation are defined, a mathematical equation can be developed to calculate the analyte concentration in an unknown sample. In some FPXRF instruments, the software of the instrument calculates the regression equation. The software uses calculated intercept and slope values to form a multiterm equation. In conjunction with the software in the instrument, the operator can adjust the multiterm equation to minimize interelement interferences and optimize the intensity calibration curve.

It is possible to define up to six linear or nonlinear terms in the regression equation. Terms can be added and deleted to optimize the equation. The goal is to produce an equation with the smallest regression error and the highest correlation coefficient. These values are automatically computed by the software as the regression terms are added, deleted, or modified. It is also possible to delete data points from the regression line if these points are significant outliers or if they are heavily weighing the data. Once the regression equation has been selected for an analyte, the equation can be entered into the software for quantitation of analytes in subsequent samples. For an empirical calibration to be acceptable, the regression equation for a specific analyte should have a correlation coefficient of 0.98 or greater or meet the DQOs of the project.

In an empirical calibration, one must apply the DQOs of the project and ascertain critical or action levels for the analytes of interest. It is within these concentration ranges or around these action levels that the FPXRF instrument should be calibrated most accurately. It may not be possible to develop a good regression equation over several orders of analyte concentration.

10.4 Compton Normalization Method: The Compton normalization method is based on analysis of a single, certified standard and normalization for the Compton peak. The Compton peak is produced from incoherent backscattering of x-ray radiation from the excitation source and is present in the spectrum of every sample. The Compton peak intensity changes with differing matrices. Generally, matrices dominated by lighter elements produce a larger Compton peak, and those dominated by heavier elements produce a smaller Compton peak. Normalizing to the Compton peak can reduce problems with varying matrix effects among samples. Compton normalization is similar to the use of internal standards in organics analysis. The Compton normalization method may not be effective when analyte concentrations exceed a few percent.

The certified standard used for this type of calibration could be an NIST SRM such as 2710 or 2711. The SRM must be a matrix similar to the samples and must contain the analytes of interests at concentrations near those expected in the samples. First, a response factor has to be determined for each analyte. This factor is calculated by dividing the net peak intensity by the analyte concentration. The net peak intensity is gross intensity corrected for baseline interference. Concentrations of analytes in samples are then determined by multiplying the baseline corrected analyte signal intensity by the normalization factor and by the response factor. The normalization factor is the quotient of the baseline corrected Compton K_α peak intensity of the SRM divided by that of the samples. Depending on the FPXRF instrument used, these calculations may be done manually or by the instrument software.

11.0 PROCEDURE

11.1 Operation of the various FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, one should consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help alleviate drift or energy calibration problems later on in analysis.

11.2 Each FPXRF instrument should be operated according to the manufacturer's recommendations. There are two modes in which FPXRF instruments can be operated: in situ and intrusive. The in situ mode involves analysis of an undisturbed soil sediment or sample. Intrusive analysis involves collection and preparation of a soil or sediment sample before analysis. Some FPXRF instruments can operate in both modes of analysis, while others are designed to operate in only one mode. The two modes of analysis are discussed below.

11.3 For in situ analysis, one requirement is that any large or nonrepresentative debris be removed from the soil surface before analysis. This debris includes rocks, pebbles, leaves, vegetation, roots, and concrete. Another requirement is that the soil surface be as smooth as possible so that the probe window will have good contact with the surface. This may require some leveling of the surface with a stainless-steel trowel. During the study conducted to provide data for this method, this modest amount of sample preparation was found to take less than 5 minutes per sample location. The last requirement is that the soil or sediment not be saturated with water. Manufacturers state that their FPXRF instruments will perform adequately for soils with moisture contents of 5 to 20 percent but will not perform well for saturated soils, especially if ponded water exists on the surface. Another recommended technique for *in situ* analysis is to tamp the soil to increase soil density and compactness for better repeatability and representativeness. This condition is especially important for heavy element analysis, such as barium. Source count times for in situ analysis usually range from 30 to 120 seconds, but source count times will vary among instruments and depending on required detection limits.

11.4 For intrusive analysis of surface or sediment, it is recommended that a sample be collected from a 4- by 4-inch square that is 1 inch deep. This will produce a soil sample of approximately 375 grams or 250 cm^3, which is enough soil to fill an 8-ounce jar. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying. The homogenization technique to be used after drying is discussed in Section 4.2. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag. One way to monitor homogenization when the sample is kneaded in a plastic bag is to add sodium fluorescein dye to the sample. After the moist sample has been homogenized, it is examined under an ultraviolet light to assess the distribution of sodium fluorescein throughout the sample. If the fluorescent dye is evenly distributed in the sample, homogenization is considered complete; if the dye is not evenly distributed, mixing should continue until the sample has been thoroughly homogenized. During the study conducted to provide data for this method, the homogenization procedure using the fluorescein dye required 3 to 5 minutes per sample. As demonstrated in Sections 13.5 and 13.7, homogenization has the greatest impact on the reduction of sampling variability. It produces little or no contamination. Often, it can be used without the more labor intensive steps of drying, grinding, and sieving given in Sections 11.5 and 11.6. Of course, to achieve the best data quality possible all four steps must be followed.

11.5 Once the soil or sediment sample has been homogenized, it should be dried. This can be accomplished with a toaster oven or convection oven. A small aliquot of the sample (20 to 50 grams) is placed in a suitable container for drying. The sample should be dried for 2 to 4 hours in the convection or toaster oven at a temperature not greater than 150°C. Microwave drying is not a recommended procedure. Field studies have shown that microwave drying can increase variability between the FPXRF data and confirmatory analysis. High levels of metals in a sample can cause arcing in the microwave oven, and sometimes slag forms in the sample. Microwave oven drying can also melt plastic containers used to hold the sample.

11.6 The homogenized dried sample material should be ground with a mortar and pestle and passed through a 60-mesh sieve to achieve a uniform particle size. Sample grinding should continue until at least 90 percent of the original sample passes through the sieve. The grinding step normally takes an average of 10 minutes per sample. An aliquot of the sieved sample should then be placed in a 31.0-mm polyethylene sample cup (or equivalent) for analysis. The sample cup should be one-half to three-quarters full at a minimum. The sample cup should be covered with a 2.5 µm Mylar (or equivalent) film for analysis. The rest of the soil sample should be placed in a jar, labeled, and archived for possible confirmation analysis. All equipment including the mortar, pestle,

and sieves must be thoroughly cleaned so that any cross-contamination is below the MDLs of the procedure or DQOs of the analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

Most FPXRF instruments have software capable of storing all analytical results and spectra. The results are displayed in parts per million and can be downloaded to a PC, which can provide a hard copy printout. Individual measurements that are smaller than three times their associated SD should not be used for quantitation.

13.0 METHOD PERFORMANCE

13.1 This section discusses four performance factors, field-based method detection limits, precision, accuracy, and comparability to EPA-approved methods. The numbers presented in Tables 4 through 9 were generated from data obtained from six FPXRF instruments. The soil samples analyzed by the six FPXRF instruments were collected from two sites in the United States. The soil samples contained several of the target analytes at concentrations ranging from nondetect to tens of thousands of mg/kg.

13.2 The six FPXRF instruments included the TN 9000 and TN Lead Analyzer manufactured by TN Spectrace; the X-MET 920 with a SiLi detector and X-MET 920 with a gas-filled proportional detector manufactured by Metorex, Inc.; the XL Spectrum Analyzer manufactured by Niton; and the MAP Spectrum Analyzer manufactured by Scitec. The TN 9000 and TN Lead Analyzer both have a HgI_2 detector. The TN 9000 utilized an Fe-55, Cd-109, and Am-241 source. The TN Lead Analyzer had only a Cd-109 source. The X-Met 920 with the SiLi detector had a Cd-109 and Am-241 source. The X-MET 920 with the gas-filled proportional detector had only a Cd-109 source. The XL Spectrum Analyzer utilized a silicon pin-diode detector and a Cd-109 source. The MAP Spectrum Analyzer utilized a solid-state silicon detector and a Cd-109 source.

13.3 All data presented in Tables 4 through 9 were generated using the following calibrations and source count times. The TN 9000 and TN Lead Analyzer were calibrated using fundamental parameters using NIST SRM 2710 as a calibration check sample. The TN 9000 was operated using 100, 60, and 60 second count times for the Cd-109, Fe-55, and Am-241 sources, respectively. The TN Lead analyzer was operated using a 60 second count time for the Cd-109 source. The X-MET 920 with the Si(Li) detector was calibrated using fundamental parameters and one well characterized site-specific soil standard as a calibration check. It used 140 and 100 second count times for the Cd-109 and Am-241 sources, respectively. The X-MET 920 with the gas-filled proportional detector was calibrated empirically using between 10 and 20 well characterized site-specific soil standards. It used 120 second times for the Cd-109 source. The XL Spectrum Analyzer utilized NIST SRM 2710 for calibration and the Compton peak normalization procedure for quantitation based on 60 second count times for the Cd-109 source. The MAP Spectrum Analyzer was internally calibrated by the manufacturer. The calibration was checked using a well-characterized site-specific soil standard. It used 240 second times for the Cd-109 source.

13.4 Field-Based Method Detection Limits: The field-based method detection limits are presented in Table 4. The field-based method detection limits were determined by collecting ten replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected method detection limits. Based on these ten replicate measurements, a standard deviation on the replicate analysis was calculated. The method detection limits presented in Table 4 are defined as 3 times the standard deviation for each analyte.

The field-based method detection limits were generated by using the count times discussed earlier in this section. All the field-based method detection limits were calculated for soil samples that had been dried and ground and placed in a sample cup with the exception of the MAP Spectrum Analyzer. This instrument can only be operated in the in situ mode, meaning the samples were moist and not ground.

Some of the analytes such as cadmium, mercury, silver, selenium, and thorium were not detected or only detected at very low concentrations such that a field-based method detection limit could not be determined. These analytes are not presented in Table 4. Other analytes such as calcium, iron, potassium, and titanium were only found at high concentrations (thousands of mg/kg) so that reasonable method detection limits could not be calculated. These analytes also are not presented in Table 4.

13.5 Precision Measurements: The precision data is presented in Table 5. Each of the six FPXRF instruments performed 10 replicate measurements on 12 soil samples that had analyte concentrations ranging from nondetects to thousands of mg/kg. Each of the 12 soil samples underwent 4 different preparation techniques from in situ (no preparation) to dried and ground in a sample cup. Therefore, there were 48 precision data points for five of the instruments and 24 precision points for the MAP Spectrum Analyzer. The replicate measurements were taken using the source count times discussed at the beginning of this section.

For each detectable analyte in each precision sample a mean concentration, standard deviation, and RSD was calculated for each analyte. The data presented in Table 5 is an average RSD for the precision samples that had analyte concentrations at 5 to 10 times the MDL for that analyte for each instrument. Some analytes such as mercury, selenium, silver, and thorium were not detected in any of the precision samples so these analytes are not listed in Table 5. Some analytes such as cadmium, nickel, and tin were only detected at concentrations near the MDLs so that an RSD value calculated at 5 to 10 times the MDL was not possible.

One FPXRF instrument collected replicate measurements on an additional nine soil samples to provide a better assessment of the effect of sample preparation on precision. Table 6 shows these results. The additional nine soil samples were comprised of three from each texture and had analyte concentrations ranging from near the detection limit of the FPXRF analyzer to thousands of mg/kg. The FPXRF analyzer only collected replicate measurements from three of the preparation methods; no measurements were collected from the *in situ* homogenized samples. The FPXRF analyzer conducted five replicate measurements of the in situ field samples by taking measurements at five different points within the 4-inch by 4-inch sample square. Ten replicate measurements were collected for both the intrusive undried and unground and intrusive dried and ground samples contained in cups. The cups were shaken between each replicate measurement.

Table 6 shows that the precision dramatically improved from the in situ to the intrusive measurements. In general there was a slight improvement in precision when the sample was dried and ground. Two factors caused the precision for the in situ measurements to be poorer. The major factor is soil heterogeneity. By moving the probe within the 4-inch by 4-inch square, measurements of different soil samples were actually taking place within the square. Table 6 illustrates the dominant effect of soil heterogeneity. It overwhelmed instrument precision when the FPXRF analyzer was used in this mode. The second factor that caused the RSD values to be higher for the in situ measurements is the fact that only five versus ten replicates were taken. A lesser number of measurements caused the standard deviation to be larger which in turn elevated the RSD values.

13.6 Accuracy Measurements: Five of the FPXRF instruments (not including the MAP Spectrum Analyzer) analyzed 18 SRMs using the source count times and calibration methods given at the beginning of this section. The 18 SRMs included 9 soil SRMs, 4 stream or river sediment SRMs, 2 sludge SRMs, and 3 ash SRMs. Each of the SRMs contained known concentrations of certain target analytes. A percent recovery was calculated for each analyte in each SRM for each FPXRF instrument. Table 7 presents a summary of this data. With the exception of cadmium, chromium, and nickel, the values presented in Table 7 were generated from the 13 soil and sediment SRMs only. The 2 sludge and 3 ash SRMs were included for cadmium, chromium, and nickel because of the low or nondetectable concentrations of these three analytes in the soil and sediment SRMs.

Only 12 analytes are presented in Table 7. These are the analytes that are of environmental concern and provided a significant number of detections in the SRMs for an accuracy assessment. No data is presented for the X-MET 920 with the gas-filled proportional detector. This FPXRF instrument was calibrated empirically using site-specific soil samples. The percent recovery values from this instrument were very sporadic and the data did not lend itself to presentation in Table 7.

Table 8 provides a more detailed summary of accuracy data for one FPXRF instrument (TN 9000) for the 9 soil SRMs and 4 sediment SRMs. Table 8 shows the certified value, measured value, and percent recovery for five analytes. These analytes were chosen because they are of environmental concern and were most prevalently certified for in the SRM and detected by the FPXRF instrument. The first nine SRMs are soil and the last 4 SRMs are sediment. Percent recoveries for the four NIST SRMs were often between 90 and 110 percent for all analytes.

13.7 Comparability: Comparability refers to the confidence with which one data set can be compared to another. In this case, FPXRF data generated from a large study of six FPXRF instruments was compared to SW-846 Methods 3050 and 6010 which are the standard soil extraction for metals and analysis by inductively coupled plasma. An evaluation of comparability was conducted by using linear regression analysis. Three factors were determined using the linear regression. These factors were the y-intercept, the slope of the line, and the coefficient of determination (r^2).

As part of the comparability assessment, the effects of soil type and preparation methods were studied. Three soil types (textures) and four preparation methods were examined during the study. The preparation methods evaluated the cumulative effect of particle size, moisture, and homogenization on comparability. Due to the large volume of data produced during this study, linear regression data for six analytes from only one FPXRF instrument is presented in Table 9. Similar trends in the data were seen for all instruments.

Table 9 shows the regression parameters for the whole data set, broken out by soil type, and by preparation method. The soil types are as follows: soil 1--sand; soil 2--loam; and soil 3--silty clay. The preparation methods are as follows: preparation 1--in situ in the field; preparation 2--in situ, sample collected and homogenized; preparation 3--intrusive, with sample in a sample cup but sample still wet and not ground; and preparation 4--sample dried, ground, passed through a 40-mesh sieve, and placed in sample cup.

For arsenic, copper, lead, and zinc, the comparability to the confirmatory laboratory was excellent with r^2 values ranging from 0.80 to 0.99 for all six FPXRF instruments. The slopes of the regression lines for arsenic, copper, lead, and zinc, were generally between 0.90 and 1.00 indicating the data would need to be corrected very little or not at all to match the confirmatory laboratory data. The r^2 values and slopes of the regression lines for barium and chromium were

not as good as for the other for analytes, indicating the data would have to be corrected to match the confirmatory laboratory.

Table 9 demonstrates that there was little effect of soil type on the regression parameters for any of the six analytes. The only exceptions were for barium in soil 1 and copper in soil 3. In both of these cases, however, it is actually a concentration effect and not a soil effect causing the poorer comparability. All barium and copper concentrations in soil 1 and 3, respectively, were less than 350 mg/kg.

Table 9 shows there was a preparation effect on the regression parameters for all six analytes. With the exception of chromium, the regression parameters were primarily improved going from preparation 1 to preparation 2. In this step, the sample was removed from the soil surface, all large debris was removed, and the sample was thoroughly homogenized. The additional two preparation methods did little to improve the regression parameters. This data indicates that homogenization is the most critical factor when comparing the results. It is essential that the sample sent to the confirmatory laboratory match the FPXRF sample as closely as possible.

Section 11.0 of this method discusses the time necessary for each of the sample preparation techniques. Based on the data quality objectives for the project, an analyst must decide if it is worth the extra time required to dry and grind the sample for small improvements in comparability. Homogenization requires 3 to 5 minutes. Drying the sample requires one to two hours. Grinding and sieving requires another 10 to 15 minutes per sample. Lastly, when grinding and sieving is conducted, time must be allotted to decontaminate the mortars, pestles, and sieves. Drying and grinding the samples and decontamination procedures will often dictate that an extra person be on site so that the analyst can keep up with the sample collection crew. The cost of requiring an extra person on site to prepare samples must be balanced with the gain in data quality and sample throughput.

13.8 The following documents may provide additional guidance and insight on this method and technique:

13.8.1 Hewitt, A.D. 1994. "Screening for Metals by X-ray Fluorescence Spectrometry/Response Factor/Compton K_α Peak Normalization Analysis." *American Environmental Laboratory.* Pages 24-32.

13.8.2 Piorek, S., and J.R. Pasmore. 1993. "Standardless, In Situ Analysis of Metallic Contaminants in the Natural Environment With a PC-Based, High Resolution Portable X-Ray Analyzer." *Third International Symposium on Field Screening Methods for Hazardous Waste and Toxic Chemicals.* Las Vegas, Nevada. February 24-26, 1993. Volume 2, Pages 1135-1151.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical management for Waste Reduction* available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

16.0 REFERENCES

1. Metorex. X-MET 920 User's Manual.

2. Spectrace Instruments. 1994. Energy Dispersive X-ray Fluorescence Spectrometry: An Introduction.

3. TN Spectrace. Spectrace 9000 Field Portable/Benchtop XRF Training and Applications Manual.

4. Unpublished SITE data, recieved from PRC Environment Management, Inc.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The pages to follow contain Tables 1 through 9 and a method procedure flow diagram.

TABLE 1
INTERFERENCE FREE DETECTION LIMITS

Analyte	Chemical Abstract Series Number	Detection Limit in Quartz Sand (milligrams per kilogram)
Antimony (Sb)	7440-36-0	40
Arsenic (As)	7440-38-0	40
Barium (Ba)	7440-39-3	20
Cadmium (Cd)	7440-43-9	100
Calcium (Ca)	7440-70-2	70
Chromium (Cr)	7440-47-3	150
Cobalt (Co)	7440-48-4	60
Copper (Cu)	7440-50-8	50
Iron (Fe)	7439-89-6	60
Lead (Pb)	7439-92-1	20
Manganese (Mn)	7439-96-5	70
Mercury (Hg)	7439-97-6	30
Molybdenum (Mo)	7439-93-7	10
Nickel (Ni)	7440-02-0	50
Potassium (K)	7440-09-7	200
Rubidium (Rb)	7440-17-7	10
Selenium (Se)	7782-49-2	40
Silver (Ag)	7440-22-4	70
Strontium (Sr)	7440-24-6	10
Thallium (Tl)	7440-28-0	20
Thorium (Th)	7440-29-1	10
Tin (Sn)	7440-31-5	60
Titanium (Ti)	7440-32-6	50
Vanadium (V)	7440-62-2	50
Zinc (Zn)	7440-66-6	50
Zirconium (Zr)	7440-67-7	10

Source: References 1, 2, and 3

TABLE 2
SUMMARY OF RADIOISOTOPE SOURCE CHARACTERISTICS

Source	Activity (mCi)	Half-Life (Years)	Excitation Energy (keV)	Elemental Analysis Range	
Fe-55	20-50	2.7	5.9	Sulfur to Chromium Molybdenum to Barium	K Lines L Lines
Cd-109	5-30	1.3	22.1 and 87.9	Calcium to Rhodium Tantalum to Lead Barium to Uranium	K Lines K Lines L Lines
Am-241	5-30	458	26.4 and 59.6	Copper to Thulium Tungsten to Uranium	K Lines L Lines
Cm-244	60-100	17.8	14.2	Titanium to Selenium Lanthanum to Lead	K Lines L Lines

Source: Reference 1, 2, and 3

TABLE 3
SUMMARY OF X-RAY TUBE SOURCE CHARACTERISTICS

Anode Material	Recommended Voltage Range (kV)	K-alpha Emission (keV)	Elemental Analysis Range	
Cu	18-22	8.04	Potassium to Cobalt Silver to Gadolinium	K Lines L Lines
Mo	40-50	17.4	Cobalt to Yttrium Europium to Radon	K Lines L Lines
Ag	50-65	22.1	Zinc to Technicium Ytterbium to Neptunium	K Lines L Lines

Source: Reference 4

Notes: The sample elements excited are chosen by taking as the lower limit the same ratio of excitation line energy to element absorption edge as in Table 2 (approximately 0.45) and the requirement that the excitation line energy be above the element absorption edge as the upper limit (L2 edges used for L lines). K-beta excitation lines were ignored.

TABLE 4
FIELD-BASED METHOD DETECTION LIMITS (mg/kg)[a]

Analyte	Instrument					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	55	NR	NR	NR	NR	NR
Arsenic	60	50	55	50	110	225
Barium	60	NR	30	400	NR	NR
Chromium	200	460	210	110	900	NR
Cobalt	330	NR	NR	NR	NR	NR
Copper	85	115	75	100	125	525
Lead	45	40	45	100	75	165
Manganese	240	340	NR	NR	NR	NR
Molybdenum	25	NR	NR	NR	30	NR
Nickel	100	NR	NA	NA	NA	NR
Rubidium	30	NR	NR	NR	45	NR
Strontium	35	NR	NR	NR	40	NR
Tin	85	NR	NR	NR	NR	NR
Zinc	80	95	70	NA	110	NA
Zirconium	40	NR	NR	NR	25	NR

Source: Reference 4

[a] MDLs are related to the total number of counts taken. See Section 13.3 for count times used to generate this table.
NR Not reported.
NA Not applicable; analyte was reported but was not at high enough concentrations for method detection limit to be determined.

TABLE 5
PRECISION

Analyte	Average Relative Standard Deviation for Each Instrument at 5 to 10 Times the MDL					
	TN 9000	TN Lead Analyzer	X-MET 920 (SiLi Detector)	X-MET 920 (Gas-Filled Detector)	XL Spectrum Analyzer	MAP Spectrum Analyzer
Antimony	6.54	NR	NR	NR	NR	NR
Arsenic	5.33	4.11	3.23	1.91	12.47	6.68
Barium	4.02	NR	3.31	5.91	NR	NR
Cadmium	29.84[a]	NR	24.80[a]	NR	NR	NR
Calcium	2.16	NR	NR	NR	NR	NR
Chromium	22.25	25.78	22.72	3.91	30.25	NR
Cobalt	33.90	NR	NR	NR	NR	NR
Copper	7.03	9.11	8.49	9.12	12.77	14.86
Iron	1.78	1.67	1.55	NR	2.30	NR
Lead	6.45	5.93	5.05	7.56	6.97	12.16
Manganese	27.04	24.75	NR	NR	NR	NR
Molybdenum	6.95	NR	NR	NR	12.60	NR
Nickel	30.85[a]	NR	24.92[a]	20.92[a]	NA	NR
Potassium	3.90	NR	NR	NR	NR	NR
Rubidium	13.06	NR	NR	NR	32.69[a]	NR
Strontium	4.28	NR	NR	NR	8.86	NR
Tin	24.32[a]	NR	NR	NR	NR	NR
Titanium	4.87	NR	NR	NR	NR	NR
Zinc	7.27	7.48	4.26	2.28	10.95	0.83
Zirconium	3.58	NR	NR	NR	6.49	NR

Source: Reference 4

[a] These values are biased high because the concentration of these analytes in the soil samples was near the detection limit for that particular FPXRF instrument.
NR Not reported.
NA Not applicable; analyte was reported but was below the method detection limit.

TABLE 6
PRECISION AS AFFECTED BY SAMPLE PREPARATION

Analyte	Average Relative Standard Deviation for Each Preparation Method		
	In Situ-Field	Intrusive-Undried and Unground	Intrusive-Dried and Ground
Antimony	30.1	15.0	14.4
Arsenic	22.5	5.36	3.76
Barium	17.3	3.38	2.90
Cadmium[a]	41.2	30.8	28.3
Calcium	17.5	1.68	1.24
Chromium	17.6	28.5	21.9
Cobalt	28.4	31.1	28.4
Copper	26.4	10.2	7.90
Iron	10.3	1.67	1.57
Lead	25.1	8.55	6.03
Manganese	40.5	12.3	13.0
Mercury	ND	ND	ND
Molybdenum	21.6	20.1	19.2
Nickel[a]	29.8	20.4	18.2
Potassium	18.6	3.04	2.57
Rubidium	29.8	16.2	18.9
Selenium	ND	20.2	19.5
Silver[a]	31.9	31.0	29.2
Strontium	15.2	3.38	3.98
Thallium	39.0	16.0	19.5
Thorium	NR	NR	NR
Tin	ND	14.1	15.3
Titanium	13.3	4.15	3.74
Vanadium	NR	NR	NR
Zinc	26.6	13.3	11.1
Zirconium	20.2	5.63	5.18

Source: Reference 4

[a] These values may be biased high because the concentration of these analytes in the soil samples was near the detection limit.
ND Not detected.
NR Not reported.

TABLE 7
ACCURACY

Analyte	Instrument															
	TN 9000				TN Lead Analyzer				X-MET 920 (SiLi Detector)				XL Spectrum Analyzer			
	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec.	SD	n	Range of % Rec.	Mean % Rec	SD	n	Range of % Rec.	Mean % Rec.	SD
Sb	2	100-149	124.3	NA	--	--	--	--	--	--	--	--	--	--	--	--
As	5	68-115	92.8	17.3	5	44-105	83.4	23.2	4	9.7-91	47.7	39.7	5	38-535	189.8	206
Ba	9	98-198	135.3	36.9	--	--	--	--	9	18-848	168.2	262	--	--	--	--
Cd	2	99-129	114.3	NA	--	--	--	--	6	81-202	110.5	45.7	--	--	--	--
Cr	2	99-178	138.4	NA	--	--	--	--	7	22-273	143.1	93.8	3	98-625	279.2	300
Cu	8	61-140	95.0	28.8	6	38-107	79.1	27.0	11	10-210	111.8	72.1	8	95-480	203.0	147
Fe	6	78-155	103.7	26.1	6	89-159	102.3	28.6	6	48-94	80.4	16.2	6	26-187	108.6	52.9
Pb	11	66-138	98.9	19.2	11	68-131	97.4	18.4	12	23-94	72.7	20.9	13	80-234	107.3	39.9
Mn	4	81-104	93.1	9.70	3	92-152	113.1	33.8	--	--	--	--	--	--	--	--
Ni	3	99-122	109.8	12.0	--	--	--	--	--	--	--	--	3	57-123	87.5	33.5
Sr	8	110-178	132.6	23.8	--	--	--	--	--	--	--	--	7	86-209	125.1	39.5
Zn	11	41-130	94.3	24.0	10	81-133	100.0	19.7	12	46-181	106.6	34.7	11	31-199	94.6	42.5

Source: Reference 4

n Number of samples that contained a certified value for the analyte and produced a detectable concentration from the FPXRF instrument.
SD Standard deviation.
NA Not applicable; only two data points, therefore, a SD was not calculated.
%Rec. Percent recovery.
-- No data.

TABLE 8
ACCURACY FOR TN 9000[a]

Standard Reference Material	Arsenic			Barium			Copper			Lead			Zinc		
	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.	Cert. Conc.	Meas. Conc.	%Rec.
RTC CRM-021	24.8	ND	NA	586	1135	193.5	4792	2908	60.7	144742	149947	103.6	546	224	40.9
RTC CRM-020	397	429	92.5	22.3	ND	NA	753	583	77.4	5195	3444	66.3	3022	3916	129.6
BCR CRM 143R	--	--	--	--	--	--	131	105	80.5	180	206	114.8	1055	1043	99.0
BCR CRM 141	--	--	--	--	--	--	32.6	ND	NA	29.4	ND	NA	81.3	ND	NA
USGS GXR-2	25.0	ND	NA	2240	2946	131.5	76.0	106	140.2	690	742	107.6	530	596	112.4
USGS GXR-6	330	294	88.9	1300	2581	198.5	66.0	ND	NA	101	80.9	80.1	118	ND	NA
NIST 2711	105	104	99.3	726	801	110.3	114	ND	NA	1162	1172	100.9	350	333	94.9
NIST 2710	626	722	115.4	707	782	110.6	2950	2834	96.1	5532	5420	98.0	6952	6476	93.2
NIST 2709	17.7	ND	NA	968	950	98.1	34.6	ND	NA	18.9	ND	NA	106	98.5	93.0
NIST 2704	23.4	ND	NA	414	443	107.0	98.6	105	106.2	161	167	103.5	438	427	97.4
CNRC PACS-1	211	143	67.7	--	772	NA	452	302	66.9	404	332	82.3	824	611	74.2
SARM-51	--	--	--	335	466	139.1	268	373	139.2	5200	7199	138.4	2200	2676	121.6
SARM-52	--	--	--	410	527	128.5	219	193	88.1	1200	1107	92.2	264	215	81.4

Source: Reference 4

[a] All concentrations in milligrams per kilogram.
%Rec. Percent recovery.
ND Not detected.
NA Not applicable.
-- No data.

TABLE 9
REGRESSION PARAMETERS FOR COMPARABILITY[1]

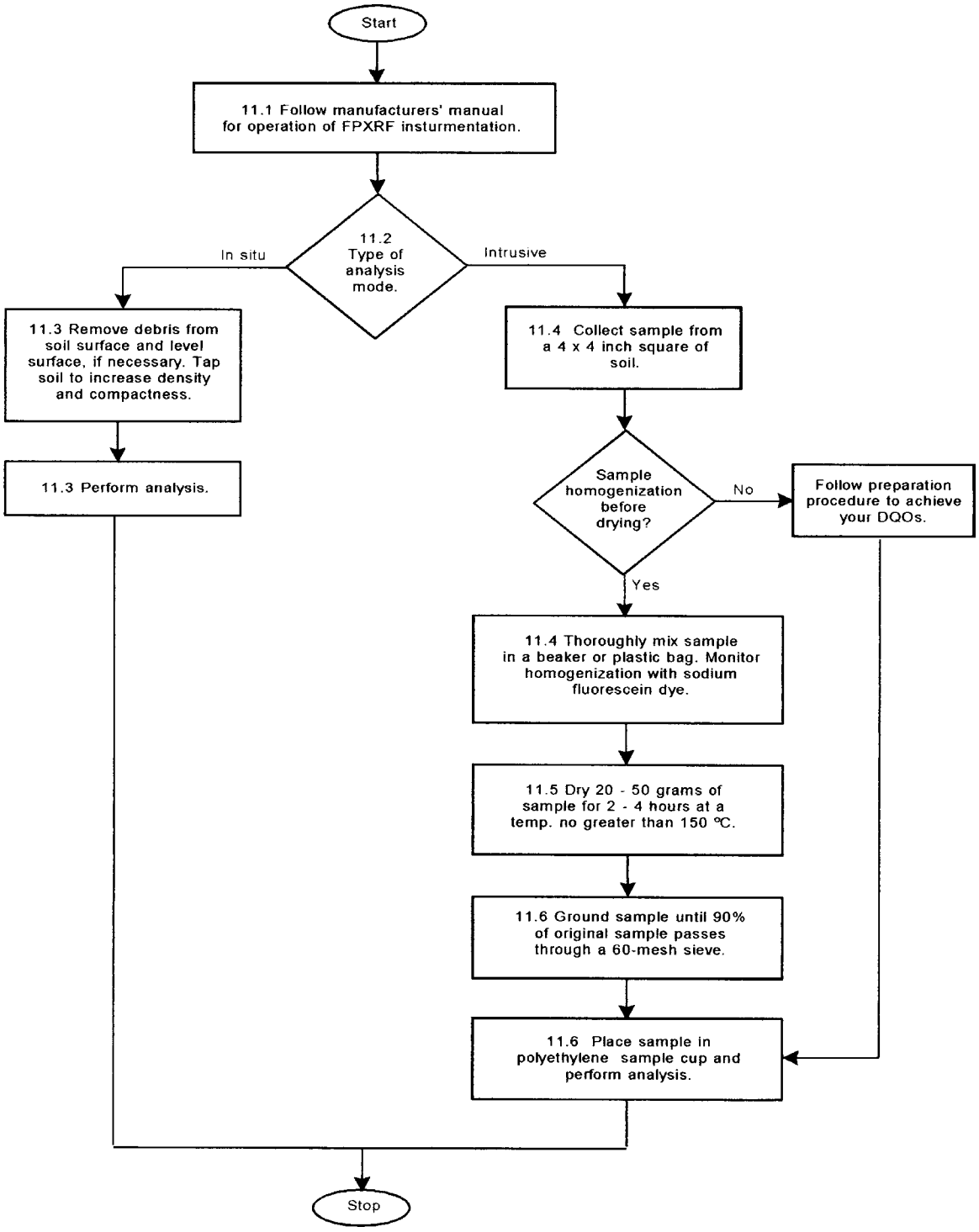
	Arsenic				Barium				Copper			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	824	0.94	1.62	0.94	1255	0.71	60.3	0.54	984	0.93	2.19	0.93
Soil 1	368	0.96	1.41	0.95	393	0.05	42.6	0.11	385	0.94	1.26	0.99
Soil 2	453	0.94	1.51	0.96	462	0.56	30.2	0.66	463	0.92	2.09	0.95
Soil 3	—	—	—	—	400	0.85	44.7	0.59	136	0.46	16.60	0.57
Prep 1	207	0.87	2.69	0.85	312	0.64	53.7	0.55	256	0.87	3.89	0.87
Prep 2	208	0.97	1.38	0.95	315	0.67	64.6	0.52	246	0.96	2.04	0.93
Prep 3	204	0.96	1.20	0.99	315	0.78	64.6	0.53	236	0.97	1.45	0.99
Prep 4	205	0.96	1.45	0.98	313	0.81	58.9	0.55	246	0.96	1.99	0.96

	Lead				Zinc				Chromium			
	n	r^2	Int.	Slope	n	r^2	Int.	Slope	n	r^2	Int.	Slope
All Data	1205	0.92	1.66	0.95	1103	0.89	1.86	0.95	280	0.70	64.6	0.42
Soil 1	357	0.94	1.41	0.96	329	0.93	1.78	0.93	—	—	—	—
Soil 2	451	0.93	1.62	0.97	423	0.85	2.57	0.90	—	—	—	—
Soil 3	397	0.90	2.40	0.90	351	0.90	1.70	0.98	186	0.66	38.9	0.50
Prep 1	305	0.80	2.88	0.86	286	0.79	3.16	0.87	105	0.80	66.1	0.43
Prep 2	298	0.97	1.41	0.96	272	0.95	1.86	0.93	77	0.51	81.3	0.36
Prep 3	302	0.98	1.26	0.99	274	0.93	1.32	1.00	49	0.73	53.7	0.45
Prep 4	300	0.96	1.38	1.00	271	0.94	1.41	1.01	49	0.75	31.6	0.56

Source: Reference 4

[1] Log-transformed data
n Number of data points
r^2 Coefficient of determination
Int. Y-intercept
— No applicable data

FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY FOR THE DETERMINATION OF ELEMENTAL CONCENTRATIONS IN SOIL AND SEDIMENT



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