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REVISION/CHANGE LOG

Revision/ Change Letter	Description of Changes	Pages Affected	Date of Revision/ Change	Approved By (signature on file)
FR0	Revision-Procedure was initially CP4-ES-5004 but is used by multiple functional areas so is being revised to a CP3 procedure. Addressed CAPA #AI-0005910 making NOTE in 6.1.8 E.1 into a step (5.1.8 F.3) and reformatting 6.1.8 (now 5.1.8) correcting second substep labeled as A (Preparation of Samples) into a B. Deleted Scientist Responsibility and included it under Sample Management Office Responsibilities.	All	4/6/2022	Documentation on file
FR0A	Periodic Review has been completed with no changes identified in procedure technical content. Nonintent change to FA, SME, SMA, Approver and dates have been incorporated per CP3-NS-2001. Data review cycle has been reset.	All	10/4/2022	
FR0B	Intent change to address CA-003896, adding step 5.4.14 to ensure notification to Characterization that data package is ready for assessment and notification to CCID group and Characterization that data has been loaded into OREIS and to address CA-004265, rewording step 5.1.7 and adding <i>Attachment 1/Laboratory Controls to Ensure Independence of Samples is Maintained</i> to Appendix B clarifying how NCS controls are incorporated into offsite lab contracts.	4,6,8,10, 12	11/22/2022	
FR1	Update procedure to address CA-005230 and general revisions	All	11/26/2024	
FR2	Update procedure to address CA-005369 which adds project responsibility for data usability in the data assessment review process.	All	6/25/2025	Documentation on file

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1.0 PURPOSE AND SCOPE

1.1 Purpose

This procedure describes the process for the following activities involving the collection of samples at the U.S. Department of Energy (DOE) owned Paducah site.

- Laboratory Statement of Work (SOW) Development
- Laboratory Contracting
- Custody of Samples and Sample Documentation
- Tracking Sample Shipments and Analysis
- Sample Receipt and Data Verification/Assessment
- Sample Returns

1.2 Scope

This procedure shall be used by the Paducah Gaseous Diffusion Plant Deactivation and Remediation (PGDP D&R) contractor personnel and subcontractor personnel for all sampling and analysis activities at the DOE owned Paducah site. The procedure allows for flexibility in implementation for programs and projects based on data collection needs and final use of the data.

This procedure does **NOT** apply to any of the following:

- Samples collected by the Safety and Health program
- Samples collected through external agency operations, such as Kentucky Department for Environmental Protection
- Nondestructive assay (NDA) measurements
- Process technology samples
- Environmental dosimetry data
- Geotechnical data

2.0 REFERENCES

2.1 Use References

- CP3-ES-2700, *Sample and Miscellaneous Data Forms*
- CP3-ES-2709, *Chain-of-Custody Forms, Sample Labels, and Custody Seals*
- CP3-ES-5003, *Quality Assured Data*
- CP3-WM-1036, *Nuclear Criticality Safety Implementation Requirements for Handling and Storage of Fissile and Potentially Fissile Waste*
- CP3-WM-1037, *Generation and Temporary Storage of Waste Materials*
- CP3-WM-3015, *Waste Packaging*
- CP3-WM-9503, *Off-Site Shipments by Air Transport*

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- DOE/LX/07-2498&D1, *Paducah Gaseous Diffusion Plant Data Management Plan*

2.2 Source References

- CP2-ES-0006, *Environmental Monitoring Plan Fiscal Year 2025 Paducah Gaseous Diffusion Plant, Paducah, Kentucky*
- CP2-ES-0103, *Environmental Radiation Protection Program for the Paducah Gaseous Diffusion Plant, Paducah, Kentucky*
- CP3-ES-0043, *Temperature Control for Sample Storage*
- CP3-ES-1034, *Nuclear Criticality Safety Requirements for Sample Labeling, Handling and Assay Smears*
- CP4-ES-2704, *Trip, Equipment, and Field Blank Preparation*
- Federal Register, 40 Code of Federal Regulations Part 136.3
- NCSR-FRNP-17-001, *Addressing Common Mode Failures of Independent Samples Sent Offsite for Analysis*

3.0 COMMITMENTS

- NCSE GEN-01, *Nuclear Criticality Safety Evaluation for General Limits Used at the Paducah Gaseous Diffusion Plant*
- NCSE 111, *Characterization of Independent Samples in the C-709 and C-710 Laboratory Facilities*

4.0 RESPONSIBILITIES

4.1 Project Team

- 4.1.1 Submits request to Sample Management Office (SMO) for collection of samples.
- 4.1.2 Coordinates sample collection and analysis with the SMO.
- 4.1.3 Assigns Project Reviewer to participate in data assessment review process.

4.2 SMO Manager

- 4.2.1 Serves as the primary contact for all matters relating to the analytical laboratories.
- 4.2.2 Ensures long-term electronic storage of data.
- 4.2.3 Ensures compliance with DOE/LX/07-2498&D1, *Paducah Gaseous Diffusion Plant Data Management Plan*.
- 4.2.4 Maintains tracking system for samples.

4.3 SMO

- 4.3.1 Creates laboratory SOW.

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- 4.3.2 Contracts laboratory services with assistance from PGDP D&R Procurement.
- 4.3.3 Performs loading of laboratory Electronic Data Deliverables (EDDs) to the Paducah Project Environmental Measurements System (PEMS).
- 4.3.4 Performs electronic verification of data using queries in PEMS.
- 4.3.5 Performs data verification steps including contractual screen.
- 4.3.6 Coordinates data validation services when requested by the project team.
- 4.3.7 Prepares project data assessment package (DAP).

4.4 Data Reviewer

- 4.4.1 Reviews project DAP and laboratory data packages.
- 4.4.2 Performs data assessment.

4.5 Project Reviewer

- 4.5.1 Reviews project DAP.
- 4.5.2 Performs data usability assessment.
- 4.5.3 Determines if quality assured data is generated and determines if data is acceptable for decision making.

NOTE:

In this procedure, Quality Assurance (QA) Reviewer does **NOT** pertain to QA personnel.

4.6 QA Reviewer

- 4.6.1 Reviews project DAP.
- 4.6.2 Performs QA review.
- 4.6.3 Verifies completion of data assessment review process.

4.7 Sampler

- 4.7.1 Ensures collection and delivery of samples to appropriate laboratory.
- 4.7.2 Ensures that sample data forms and chain-of-custody (COC) forms are complete.
- 4.7.3 Communicates final disposition of “hold samples” or returned samples to the SMO.

5.0 GENERAL INFORMATION

None

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6.0 INSTRUCTIONS

6.1 Laboratory SOW Development

Project Team

- 6.1.1 Provide the SMO with analytical requests using an appropriate template [Quality Assurance Project Plan (QAPP), Sample Analysis Plan (SAP), Sample Analysis & Event Plan (SAEP), CP3-ES-1034-F01, *Sample Request Form*, and/or e-mail containing pertinent information related to sampling analyses and requirements].
- 6.1.2 Coordinate sample collection and analysis with the SMO.
- 6.1.3 Instruct project personnel that questions relating to sample results and sample material should be vetted through the SMO.
- 6.1.4 Ensure that CP3-ES-5003, *Quality Assured Data*, is followed throughout the data collection activity to ensure data quality.

SMO

- 6.1.5 Log the project into PEMS.
- 6.1.6 Prepare the laboratory SOW based on the analytical request information received from the project team (i.e., QAPP, SAP, SAEP, CP3-ES-1034-F01, and/or e-mail).
- 6.1.7 Create a laboratory SOW file and add all pertinent information (i.e. laboratory SOW, analytical request information, etc.).

NOTES:

Independent Samples: Samples which are capable of providing the value of a parameter with **NO** single point failure which would invalidate the results.

Independent Analysts: Two different individual analysts that, while performing analyses, do **NOT** rely upon observed actions of/or assist the other individual analyst performing sample preparation, analysis, calculation, data entry, or review of the independent sample **or** one analyst that completes sample preparation, analysis, calculation, data entry, and review of first "A" sample before starting second "B" sample on different days.

- 6.1.8 **If** preparing a laboratory SOW for Nuclear Criticality Safety (NCS) assay smears or NCS bulk assay samples, **then** ensure *Attachment 1/Laboratory Controls to Ensure Independence of Samples is Maintained* identified in Appendix B is attached to the laboratory SOW.

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- 6.1.9 Specify the quality control samples (such as trip blank, field blanks, equipment rinsate blanks, or field duplicates) that are identified in project QAPP, SAP, or SAEP in the laboratory SOW.
- 6.1.10 Provide laboratory SOW to project team for review.

6.2 Laboratory Contracting

- 6.2.1 Determine the analytical laboratory to be used.
- 6.2.2 Provide laboratory SOW to laboratory for review and approval.

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- 6.2.3 Provide additional information requested by the laboratory, if applicable.
- 6.2.4 Request the sample container and preservative requirements from laboratory.
- 6.2.5 Request sample containers with appropriate preservatives to be shipped from laboratory.

6.3 Custody of Samples and Sample Documentation

Sampler

NOTE:

Deactivated chains for “hold samples” are to document the disposal path for the sample.

- 6.3.1 Ensure that COC forms for samples, including “hold samples”, are properly completed (or deactivated) according to CP3-ES-2709, *Chain-of-Custody Forms, Sample Labels, and Custody Seals*.
- 6.3.2 Ensure that sample data forms are properly completed according to CP3-ES-2700, *Sample and Miscellaneous Data Forms*.

NOTE:

Proper radioactive surveys and approval from PGDP D&R contractor Transportation group may be required prior to shipping samples off-site.

- 6.3.3 **If applicable, then** coordinate shipment or delivery of samples to the appropriate laboratory according to CP3-WM-9503, *Off-site Shipments by Air Transport*.
- 6.3.4 Retain any samples that are collected and **NOT** submitted to a laboratory (“hold samples”) using proper preservation and storage conditions until notified by the SMO for decision on additional analysis.

NOTES:

Samples are **NOT** considered or managed as waste by way of a Resource Conservation Recovery Act (RCRA) exemption until determined to be **NO** longer needed.

A disposal path for “hold samples” is required in advance to ensure that samples are managed quickly and compliantly.

- 6.3.5 Consult with Waste Engineer and/or Regulatory Compliance for proper waste disposal/storage of “hold samples” in advance to establish a proper disposal path.
- 6.3.6 **If** “hold samples” are no longer needed, **then** dispose of “hold samples” according to regulatory compliance guidelines.

6.4 Sample Receipt and Data Verification

SMO

- 6.4.1 Ensure the laboratory received the samples.
- 6.4.2 Log the date that the sample was received by the laboratory into PEMS.

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- 6.4.3 Check laboratory sample receipt confirmation report for accuracy **and** notify laboratory about any discrepancies found.
- 6.4.4 Track the receipt of EDDs and laboratory data packages.
- 6.4.5 Complete data verification steps according to CP3-ES-5003.
- 6.4.6 **If** data validation is required, **then** coordinate data validation services according to CP3-ES-5003.
- 6.4.7 Create the project DAP.
- 6.4.8 Provide project DAP and laboratory data packages to Data Reviewer.

6.5 Data Assessment & Data Usability Assessment

Data Reviewer

- 6.5.1 Perform data assessment according to CP3-ES-5003.
- 6.5.2 Notify the SMO when data assessment is complete.

Project Reviewer

- 6.5.3 Perform data usability assessment according to CP3-ES-5003.
- 6.5.4 Notify the SMO when data usability assessment is complete.

SMO

- 6.5.5 Ensure all comments or issues identified during data assessment and data usability assessment have been resolved.
- 6.5.6 Notify QA Reviewer that project DAP is ready for QA review.

6.6 QA Review

QA Reviewer

- 6.6.1 Perform QA review according to CP3-ES-5003.
- 6.6.2 Notify the SMO when QA review is complete.

6.7 Data Classification Review

SMO

- 6.7.1 **If** project DAP contains data that is of non-environmental matrices (i.e., waste or characterization projects), **then** submit project DAP for a Derivative Classifier review according to CP3-ES-5003.
- 6.7.2 Electronically load analytical data from PEMS into Paducah Oak Ridge Environmental Information System (OREIS).

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- 6.7.3 Send OREIS report and Excel file of analytical data to Project Team.
- 6.7.4 **If** project data contains component identification number assigned by Characterization and Criticality Incredible Database (CCID), **then** send OREIS report, Excel file of analytical data, and a completed project DAP to the CCID group.
- 6.7.5 Submit completed project DAP and applicable laboratory data packages to Records Management.

6.8 Sample Returns

- 6.8.1 Utilize a tracking system to denote if a sample is to be disposed of by the laboratory **or** if the sample is to be returned to PGDP D&R contractor.
- 6.8.2 Provide the Project Team, Samplers, and Waste Generator Manager with a listing of samples to be returned.
- 6.8.3 **If** the project is considered closed, **then** contact Waste Generator Manager for direction on disposal of samples.

NOTE:

Approval is to be obtained from Project Team, Samplers, and Waste Generator Manager before samples can be returned.

- 6.8.4 Notify the laboratory that samples are ready for return.

Sampler

NOTES:

Upon return of samples, samples will be managed as waste by the Project Team and Samplers according to CP3-WM-1037, *Generation and Temporary Storage of Waste Materials*, CP3-WM-3015, *Waste Packaging* and CP3-WM-1036, *Nuclear Criticality Safety Implementation Requirements for Handling and Storage of Fissile and Potentially Fissile Waste* (if samples are fissile).

Samples shipped to a laboratory are **NOT** candidates for RCRA waste consideration via an exemption.

A disposal path for samples returning from the laboratory is required in advance to ensure that samples are managed quickly and compliantly.

- 6.8.5 Receive the sample shipment from the laboratory **and** notify Project Team of sample shipment receipt.
- 6.8.6 Notify the SMO of the sample shipment receipt **and** final disposition of the samples.

SMO

- 6.8.7 Document in tracking system and/or PEMS that samples have been received from the laboratory **and** applicable disposition information.

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6.8.8 Confirm that all samples associated with the project have been denoted in the tracking system and/or PEMS as follows:

- Disposed and/or consumed by the laboratory
- Disposed by PGDP D&R contractor as a returned sample
- Disposed by PGDP D&R contractor as a “hold sample”

7.0 RECORDS

7.1 Records Generated

The following records may be generated by this procedure:

- Laboratory SOW files
- Project DAP

Forms are to be completed according to CP3-OP-0024, *Forms Control*.

7.2 Records Disposition

The records are to be maintained according to CP3-RD-0010, *Records Management Process*.

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Appendix A–Acronyms/Definitions

ACRONYMS

CCID – Characterization and Criticality Incredible Database

COC – Chain of Custody

DAP – Data Assessment Package

DOE – U.S. Department of Energy

EDD – Electronic Data Deliverable

NCS – Nuclear Criticality Safety

NCSE – Nuclear Criticality Safety Evaluation

OREIS – Paducah Oak Ridge Environmental Information System

PEMS – Paducah Project Environmental Measurements System

PGDP D&R – Paducah Gaseous Diffusion Plant Deactivation and Remediation

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

RCRA – Resource Conservation Recovery Act

SAEP – Sample Analysis and Event Plan

SAP – Sample Analysis Plan

SMO – Sample Management Office

SOW – Statement of Work

DEFINITIONS

Contractual Screen – A process of evaluating a set of data against the requirements specified in the laboratory SOW to ensure that all requested information is received. The contractual screen includes, but is **NOT** limited to, the review of COC information, analytes requested, method used, units, holding times, and reporting limits achieved.

Data Reviewer – Performs independent review of data presented in project DAP. Data Reviewer can be personnel from SMO or Characterization organizations who are appropriately trained. Data Reviewer and QA Reviewer cannot be the same individual.

Hold Samples – Samples that are collected for the purpose of potential analysis, generally dependent upon the results of a preceding screening sample. Hold samples are collected and retained using proper preservation and are tracked in PEMS as any other active sample unless disposed.

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Appendix A–Acronyms/Definitions (continued)

Independent Analysts – Two different individual analysts that, while performing analyses, do **NOT** rely upon observed actions of/or assist the other individual analyst performing sample preparation, analysis, calculation, data entry, or review of the independent sample **or** one analyst that completes sample preparation, analysis, calculation, data entry, and review of first “A” sample before starting second “B” sample on different days.

Independent Samples – Samples which are capable of providing the value of a parameter with no single point failure which would invalidate the results.

PEMS – The data management system that supports the project’s sampling and data management activities. The system tracks sampling requests and projects through assignment of a project identification code (i.e., ProjectID) along with charge code, sampling timeframe, status, etc. The system also generates COC forms, sample container labels, and sample data forms. The system tracks sampling progress and stores project specific data.

Project Reviewer – Performs independent review of data presented in project DAP. Project Reviewer is assigned by the project team and can be personnel from project team who are appropriately trained. The Project Reviewer bears the ultimate responsibility for determining the usability of a data set for decision making purposes. Project Reviewer and QA Reviewer cannot be the same individual.

Project Team – The project team consists of project personnel responsible for initiating a data collection activity (i.e., sampling event). The project team defines the project DQOs and submits request to the SMO for collection of samples. The project team coordinates sample collection and analysis with the SMO to ensure project requirements are met. The project team assigns a representative of the project to serve as the Project Reviewer.

QA Reviewer – Performs independent review of project DAP and verifies completion of data assessment. QA Reviewer is a member of the SMO who is appropriately trained. QA Reviewer and Data Reviewer cannot be the same individual.

Appendix B—Attachment 1/Laboratory Controls to Ensure Independence of Samples is Maintained

Attachment 1

Laboratory Controls to Ensure Independence of Samples is Maintained

Independent Samples: Samples which are capable of providing the value of a parameter with no single point failure which would invalidate the results.

Independent analysts: Two different individual analysts that, while performing analyses, do not rely upon observed actions of /or assist the other individual analyst performing sample preparation, analysis, calculation, data entry or review of the independent sample **OR** one analyst that completes sample preparation, analysis, calculation, data entry, and review of first "A" sample before starting second "B" sample on different day.

Sample Receipt

Ensure both samples have been assigned one unique customer sample number. One of the samples will be identified with an "A" designator and the independent sample will be identified with a "B" designator. If samples are not labeled properly, do not analyze; a new sample is required.

Preparation of Samples

The samples with an "A" designator will be prepared by an analyst who is independent from the analyst who prepares the samples with a "B" designator. If a single analyst prepares both sets of samples, then the samples with an "A" designator will be prepared on a different day from the samples with a "B" designator.

Analyzing Samples

If complete sample is not used for analysis (e.g., smear) then ensure a representative aliquot is used by homogenizing sample material before aliquot is taken for analysis.

Ensure A and B samples are analyzed on different instruments by different analysts **or**:

- 1) If both "A" and "B" samples are analyzed on a single instrument, ensure analysis is performed on different days or by different analysts.
- 2) If both "A" and "B" samples are analyzed by a single analyst, ensure the samples are analyzed on different days and that result from the first analysis is entered into a LIMS system before performing the second analysis.

Instrument Controls:

- 1) Ensure calibration standards are not from same parentage as control (calibration verification standards).
- 2) Ensure appropriate QC samples (e.g., blanks, duplicates, spikes) are analyzed with each batch.
- 3) Using a standard (e.g., CCV) ensure laboratory instrument response is within 3 sigma of the calibrated instrument response **before** and **following** each measurement session.
- 4) Ensure "A" and "B" samples are not analyzed in the same measurement session.
- 5) Do not consider the results of an analysis valid until the final check of instrument has been satisfactorily completed with a standard.
- 6) Analysis results that are below MDA or specified standard 1 level are reported as nondetects and a weight percent for U-235 is not reported if U-235 is nondetect.
- 7) The total propagated error (1.96 sigma) will be determined and reported for each result.
- 8) Follow guidelines specified in white paper titled "Modification for the Calculation for wt% U-235" signed by FRNP on 3/24/2022.

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**Appendix B–Attachment 1/Laboratory Controls to Ensure Independence of Samples is Maintained
(Continued)**

Attachment 1

Data Approval

Ensure review/approval of data entry into the LIMS database is performed by an independent person not responsible for analyzing the sample.

Data Reporting

Verbal relay of the analytical results is prohibited. If sample results are manually entered into the LIMS database, ensure results are verified to be correctly entered by an independent person.

Each result will have a TPU value reported (1.96 sigma).

If the U-234 concentration or the U-236 concentration in the sample is below the standard 1 levels, then do not use that isotope concentration in the wt% U-235 calculation but still report a wt% U-235 result.

If the U-235 concentration or the U-238 concentration in the sample is below the standard 1 levels, then report zero for wt% U-235 with U qualifier.

NCS Approval: Joseph Kowen / Joe Nelson / 32731 / 24 May 22

NCSR Reference: NCSR-RR.NP-17-001