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<b>CP3-ES-5004 FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	Page 1 of 13
<b>DOCUMENT CATEGORY:</b> Administrative		
<b>LEVEL OF USE:</b> Information Level		
<b>FUNCTIONAL AREA:</b> Sample Management Office		<b>SUBJECT MATTER EXPERT:</b> Lisa Crabtree, Sample Management Office Manager
<b>SUBJECT MATTER AREA:</b> Sample Management Office		
<b>NUCLEAR SAFETY REVIEW DOCUMENTATION:</b> FRNP-22-0829-S, Rev 0		<b>APPROVED BY/DATE</b> (Signature on file): Jolie Fleming, Technical Services Director 11/22/2022
<b>REQUIRED REVIEW DATE</b> (or expiration date for temporary change): 10/4/2025		<b>EFFECTIVE DATE:</b> 11/28/2022

<b>REVISION/CHANGE LOG</b>			
Revision/Change Letter	Description of Changes	Pages Affected	Date of Revision/Change
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
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

**TABLE OF CONTENTS**

1.0 PURPOSE AND SCOPE ..... 3

    1.1 Purpose..... 3

    1.2 Scope..... 3

2.0 REFERENCES..... 3

    2.1 Use References..... 3

    2.2 Source References..... 3

3.0 COMMITMENTS..... 4

4.0 RESPONSIBILITIES..... 4

    4.1 Project Manager ..... 4

    4.2 Data Reviewer..... 4

    4.3 Quality Reviewer ..... 5

    4.4 Sampler ..... 5

    4.5 SMO..... 5

5.0 INSTRUCTIONS..... 5

    5.1 SOW Development ..... 5

    5.2 Laboratory Contracting..... 6

    5.3 Custody of Samples and Sample Documentation ..... 6

    5.4 Sample Receipt and Data Verification/Assessment ..... 7

    5.5 Sample Returns ..... 8

6.0 RECORDS ..... 9

    6.1 Records Generated ..... 9

    6.2 Records Disposition ..... 9

APPENDIX A – ACRONYMS/DEFINITIONS ..... 10

APPENDIX A – ACRONYMS/DEFINITIONS (CONTINUED) ..... 11

APPENDIX B – ATTACHMENT 1/LABORATORY CONTROLS TO ENSURE INDEPENDENCE OF  
SAMPLES IS MAINTAINED ..... 12

<b>CP3-ES-5004 FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 3 of 13</b>
---------------------------------	---	---------------------

## **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.

- 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 Deactivation and Remediation (D&R) contractor personnel, and subcontractor personnel for all sampling and analysis activities at the U.S. 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.

Exceptions:

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 Environment Protection
- Nondestructive assay measurements
- Process technology samples

## **2.0 REFERENCES**

### **2.1 Use References**

- CP3-ES-2708, *Chain-of-Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*
- CP3-ES-5003, *Quality Assured Data*
- CP3-WM-1037, *Generation and Temporary Storage of Waste Materials*
- CP3-WM-3015, *Waste Packaging*
- CP3-WM-9503, *Off-Site Shipments by Air Transport*
- CP4-ES-2700, *Logbooks and Data Forms*

### **2.2 Source References**

- BJC/PAD-141, *Paducah Gaseous Diffusion Plant, Department of Energy National Emission Standards for Hazardous Air Pollutants (NESHAP) Management Plan*

<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 4 of 13</b>
---------------------------------------	---	---------------------

- CP2-ES-0006, *Environmental Monitoring Plan Paducah Gaseous Diffusion Plant, Paducah, Kentucky*
- CP2-ES-0103, *Environmental Radiation Protection Program*
- CP3-ES-0043, *Temperature Control for Sample Storage*
- CP3-ES-1034, *Nuclear Criticality Safety Requirements for Sample Labeling, Handling and Assay Smears*
- CP3-SM-0017, *Measuring and Test Equipment*
- CP3-WM-1036, *Nuclear Criticality Safety Implementation Requirements for Handling and Storage of Fissile and Potentially Fissile Waste*
- 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*

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### 3.0 COMMITMENTS

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

### 4.0 RESPONSIBILITIES

#### 4.1 Project Manager

Coordinates sample collection, sample analysis, data assessment, and decision-making. Project manager has ultimate responsibility but designated representative may include the following:

- Technical lead
- Characterization
- Compliance coordinator
- Individual that needs data to support decision- making

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#### 4.2 Data Reviewer

- 4.2.1 Performs data assessment.
- 4.2.2 Determines if quality assured data is generated.

Note:

In this procedure, Quality Reviewer does NOT pertain to Quality Assurance personnel.

### 4.3 Quality Reviewer

Reviews data to ensure that data quality requirements are met.

### 4.4 Sampler

4.4.1 Ensures collection and delivery of samples to appropriate laboratory.

4.4.2 Ensures that field records (field logbook and/or sample data forms, and chain-of-custody[COC] records) are complete.

4.4.3 Communicates final disposition of “hold samples” or returned samples to the Sample Management Office (SMO).

### 4.5 SMO

4.5.1 Ensures long-term electronic storage of data.

4.5.2 Performs loading of Electronic Data Deliverables (EDDs).

4.5.3 Performs electronic verification of data.

4.5.4 Ensures compliance with applicable Data Management Implementation Plan.

4.5.5 Maintains tracking system for samples.

4.5.6 Serves as the primary contact for all matters relating to the analytical laboratories.

4.5.7 Creates laboratory SOW.

4.5.8 Contracts laboratory services.

4.5.9 Performs contractual screenings.

4.5.10 Coordinates independent third-party data validation.

## 5.0 INSTRUCTIONS

### 5.1 SOW Development

#### Project Manager

5.1.1 Provide the SMO with analytical requests using an appropriate template (Quality Assurance Project Plan, Sample Analysis Plan, Sample Request Form, or email containing pertinent information related to sampling analyses and requirements).

5.1.2 Instruct personnel that questions relating to sample results and sample material should be vetted through the SMO.

5.1.3 Ensure that CP3-ES-5003, *Quality Assured Data*, is followed throughout the sample collection process to ensure data quality.

**SMO**

- 5.1.4 Establish Paducah Project Environmental Measurements System (PEMS) for use during sample collection, if applicable.
- 5.1.5 Log the project into the Paducah Analytical Project Tracking System.
- 5.1.6 Prepare the laboratory SOW based on analytical request received from Project Manager.

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.

5.1.7 **If** preparing an 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 SOW.

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5.1.8 Provide laboratory SOW to project for review.

**5.2 Laboratory Contracting**

- 5.2.1 Determine the analytical laboratory to be used.
- 5.2.2 Provide SOW to laboratory for review and approval.
- 5.2.3 Provide additional information requested by the laboratory, if applicable.
- 5.2.4 Request the sample container and preservative requirements from laboratory.
- 5.2.5 Request sample containers with appropriate preservatives to be shipped from laboratory.
- 5.2.6 Specify the quality control (QC) samples (such as trip blank, field blanks, equipment rinsates, or field duplicates) that are identified by using CP3-ES-5003.

**5.3 Custody of Samples and Sample Documentation**

**Sampler**

5.3.1 **If** applicable, **then** develop sampling schedule and train staff on proper procedures for sample identification, custody, and labeling.

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

5.3.2 Ensure that COC forms for samples, including “hold samples”, are properly completed (or deactivated) according to CP3-ES-2708, *Chain-of-Custody Forms, Field Sample Logs, Sample Labels, and Custody Seals*.

<b>CP3-ES-5004 FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 7 of 13</b>
---------------------------------	---	---------------------

- 5.3.3** Ensure that logbooks and/or sample data forms are properly completed according to CP4-ES-2700, *Logbooks and Data Forms*.

**NOTE:**

Proper radioactive screening tests and U.S. Department of Transportation approvals may be required prior to shipping samples off-site.

- 5.3.4** If applicable, **then** coordinate shipment or delivery of samples to the appropriate laboratory in accordance with CP3-WM-9503, *Off-site Shipments by Air Transport*.

- 5.3.5** 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.

- 5.3.6** Consult with Waste Engineer and/or regulatory compliance for proper waste disposal/storage of “hold samples” in advance to establish a proper disposal path.

- 5.3.7** If “hold samples” are no longer needed, **then** dispose of “hold samples” in accordance with regulatory compliance guidelines.

**5.4 Sample Receipt and Data Verification/Assessment**

**SMO**

- 5.4.1** Ensure the laboratory received the samples.
- 5.4.2** Log the date that the sample was received by the laboratory into Paducah PEMS.
- 5.4.3** Track the receipt of electronic data deliverables and laboratory data packages.
- 5.4.4** Electronically load analytical data from laboratory EDD into Paducah PEMS database.
- 5.4.5** Perform contractual screening on all deliverables.
- 5.4.6** If validation is required, **then** prepare a validation SOW and send laboratory data packages to the data validator.
- 5.4.7** Provide the data assessment package to the data reviewer, in order to perform data assessment.

**Data Reviewer**

- 5.4.8** Perform data assessment on the data assessment package.
- 5.4.9** Provide the data assessment package to the quality reviewer, in order to perform a Quality Assurance review.

<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 8 of 13</b>
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**Quality Reviewer**

**5.4.10** Perform a Quality Assurance review on data assessment packages as required.

**SMO**

**5.4.11** Ensure all comments or issues identified during data assessment have been resolved.

**5.4.12** Electronically load analytical data from Paducah PEMS database into Paducah Oak Ridge Environmental Information System (OREIS).

**5.4.13** Ensure all applicable emails and required forms are included in the data assessment package.

**5.4.14** **If** the analytical data is from intrusive samples, **then** perform the following:

- A.** Place data assessment package within the directory: S:\Env Services\Characterization Master\! Data Assessment Review.
- B.** Notify Characterization by emailing the characterization distribution list that the analytical data is ready for assessment.
- C.** **After** data assessment process is complete, electronically load analytical data from Paducah PEMS database into Paducah OREIS.
- D.** Send OREIS report and excel file of analytical data to Characterization and CCID group.

**5.4.15** Submit completed data assessment package and applicable laboratory data packages to Records Management.

**5.5 Sample Returns**

**5.5.1** Utilize a tracking system to denote if a sample is to be disposed by the laboratory **or** if the sample is to be returned to PGDP D&R.

**5.5.2** Provide the Project Manager, the Samplers, and the Waste Generator Manager with a listing of samples to be returned.

**5.5.3** **If** the project is considered closed, **then** contact Waste Generator Manager for direction on disposal of samples.

**NOTE:**  
 Approval is to be obtained by Project Manager, Samplers, and Waste Generator Manager before samples can be returned.

**5.5.4** Notify the laboratory that samples are ready for return.

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<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 9 of 13</b>
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## **Sampler**

### NOTE:

Upon return of samples, samples will be managed as waste by the Project Manager in accordance with CP3-WM-1037, *Generation and Temporary Storage of Waste Material* and CP3-WM-3015, *Waste Packaging*.

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

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

- 5.5.5 Receive the sample shipment and notify Project Manager.
- 5.5.6 **If** project is closed, **then** contact Waste Generator Manager for direction on disposal of samples.
- 5.5.7 Notify SMO of the sample receipt **and** final disposition of the samples.

## **SMO**

- 5.5.8 Document in Paducah PEMS that samples have been received from the laboratory **and** applicable disposition information.
- 5.5.9 Confirm that all samples associated with the project have been denoted in the tracking system and/or Paducah PEMS as follows:
  - Disposed and/or consumed by the laboratory
  - Disposed by PGDP D&R as a returned sample
  - Disposed by PGDP D&R as a “hold sample”

## **6.0 RECORDS**

### **6.1 Records Generated**

None

### **6.2 Records Disposition**

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

<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 10 of 13</b>
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## Appendix A – Acronyms/Definitions

### ACRONYMS

**CCID** – Characterization and Criticality Incredible Database

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**CCV** – Continuing Calibration Verification

**COC** – Chain of Custody

**DAP** –Data Assessment Package

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**DOE** – United States Department of Energy

**EDD** – Electronic Data Deliverable

**MDA** – Minimum Detectable Activity

**NCS** – Nuclear Criticality Safety

**OREIS** – Paducah Oak Ridge Environmental Information System

**PEMS** – Project Environmental Measurements System

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

**QA** – Quality Assurance

**QC** – Quality Control

**RCRA** – Resource Conservation Recovery Act

**SMO** – Sample Management Office

**SOW** – Statement of Work

### 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 COC, analytes requested, method used, electronic data deliverables, units, holding times, and reporting limits achieved.

**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 Paducah PEMS as any other active sample unless disposed.

**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.

<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 11 of 13</b>
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### **Appendix A – Acronyms/Definitions (Continued)**

**Paducah Analytical Project Tracking System** – A Paducah system developed to track sampling requests and projects 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 SMO to track various information about the event such as the charge number, sampling time frame, status, etc.

**Paducah PEMS** – The data management system that supports the project’s sampling and data management activities. The system generates COC forms, bottle labels, and field forms; tracks sampling progress, and stores project specific data.

<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 12 of 13</b>
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**Appendix B – Attachment 1/Laboratory Controls to Ensure Independence of Samples is Maintained**

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**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.

Page 1 of 2  
24 May 22

<b>CP3-ES-5004</b> <b>FRev. 0B</b>	<b>TITLE:</b> Sample Tracking, Lab Coordination, and Sample Handling	<b>Page 13 of 13</b>
---------------------------------------	---	----------------------

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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: *[Signature]* / Joe Nelson / 32731 / 24 May 22

NCSR Reference: NCSR-RAMP-LT-001

Page 2 of 2  
24 May 22