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**PROCEDURE FOR IDENTIFICATION AND CONTROL OF SAMPLES** 

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### **PROCEDURE FOR IDENTIFICATION AND CONTROL OF SAMPLES**

### **1.0 PURPOSE**

This quality administrative procedure (QP) describes the methods for the identification and control of samples used for, or resulting from, any scientific work or investigation conducted by Los Alamos National Laboratory (LANL) for the Yucca Mountain Project (YMP or Project).

### 2.0 SCOPE

This QP is applicable to all LANL personnel and their subcontractors who conduct scientific investigations as part of the Project. This QP applies to samples collected and/or used by LANL personnel or their subcontractors; it does not apply to the handling and control of engineered items or construction materials. Samples received from other organizations are identified and controlled by these organizations' procedures until the samples arrive at LANL.

### **3.0 REFERENCES**

3.1 Requirement Documents

LANL-YMP-Quality Assurance Program Plan (QAPP), Section 8.

#### **3.2** YMP Procedures

AP 6.2Q, Management and Operation of Sample-Handling Activities at Yucca Mountain Project Borehole Sites.

AP 6.3Q, Use of Sample Management Facility by Yucca Mountain Project Participants.

AP 6.6Q, Field Collection, Documentation, and Specimen Removal of Exploratory Shaft and Drift Rock.

BTP-SMF-001, Management and Operation of the Sample Management Facility.

BTP-SMF-002, Transport, Receipt, and Admittance for Curation of Yucca Mountain Project Borehole Samples.

BTP-SMF-003, Verification of Field Logging and Documentation of Yucca Mountain Project Core and Cuttings.

BTP-SMF-004, Physical Processing and Storage of Yucca Mountain Project Core and Cuttings.

BTP-SMF-005, Examination of Yucca Mountain Project Samples by Participants.

BTP-SMF-006, Removal of Whole Core and Other Specimens from Yucca Mountain Project Samples for Shipment and Remnant Return. BTP-SMF-007, Acceptance for Curation of Selected Yucca Mountain Project Samples and Documentation.

BTP-SMF-008, Field Logging, Handling, and Documenting Yucca Mountain Project Borehole Samples.

3.3 LANL Procedures

TWS-QAS-QP-3.5, Procedure for Documenting Scientific Investigations TWS-QAS-QP-13.1, Procedure for Handling, Storage, and Shipping TWS-QAS-QP-17.1, Procedure for the LANL Group Resident File

### 4.0 DEFINITIONS

4.1 Archive Sample

An archive sample is a specimen that is stored in the sample management facility (SMF) for future scientific investigations, confirmation tests, and experiments (i.e., stored for posterity).

4.2 Chain of Custody

Chain of custody refers to the documentation of the sequence in which samples are transferred from their source to users and custodians.

4.3 Bulk Sample

A bulk sample consists of irregularly shaped and sized rocks. These rocks may be a representative portion of the muck from each round of blasting during the excavation of the exploratory shafts (ES) and drifts or from an outcrop or surface exposure. This definition excludes core from drill holes.

4.4 Core Sample

A core sample is a cylindrical section of rock taken as a sample of the interval penetrated by a core bit and brought to the surface for examination and/or laboratory analysis.

4.5 Curation

Curation is the process of storing in a safe manner all samples, specimens, and remnants and their associated documentation.

### 4.6 Cuttings Sample

Cuttings samples are chips of rock cut by a drill bit during drilling and removed from the borehole by reverse circulation methods.

4.7 Drift

A drift is an excavation mined laterally from the ESs. The exploratory shaft facility (ESF) will contain a series of drifts.

### 4.8 Drilling Program Package

A drilling program package, which is prepared for each borehole, is a set of plans that describes the scope of work to be performed, the general and detailed requirements for performing the work, and the parameters to be used while drilling or performing work on the boreholes. The drilling program package consists of a work order, a criteria letter, a drilling program, and a cost estimate.

### 4.9 Exploratory Shafts (ES)

An ES is one of two vertically mined components of the ESF used for access to the potential emplacement horizon and for scientific investigations included in the Yucca Mountain Site Characterization Plan (SCP). Two ESs are planned for the ESF.

### 4.10 Exploratory Shaft Facility (ESF)

The ESF consists of surface structures, the two shafts, and a series of drifts and rooms in which the host rock will be studied in situ.

4.11 Muck Pile

Muck is broken rock that results from ESF excavation during mining operations.

4.12 Remnant

A remnant is the portion of a sample or specimen an investigator returns to the SMF after analysis and testing.

4.13 Sample

A sample is an item that is a part of a population whose properties are studied to gain information about the group presented for examination. Sample types handled at the SMF include, but are not limited to, surface and subsurface rocks and fluids, drill core, gases, and biota. Examples of samples covered by this procedure include whole core, research split core, cuttings, bulk material, muck, hand samples, water, and other geologic samples collected at YMP field sites. Although "sample" is often used interchangeably with "specimen," the terms are used in this document as defined in this section.

#### 4.14 Specimen

A specimen is a portion of a sample that has been removed and that will be given a unique identification number to permit tracking. A specimen can be synonymous with "split."

### 4.15 Sample Management Facility (SMF)

The SMF consists of a physical facility and equipment designed to log in, store, and distribute samples collected at the Yucca Mountain site. It is also the repository for remnants returned by investigators for bulk samples to be used for continuing studies and tests and for archive samples kept for future scientific investigations. The SMF is operated by the Technical and Management Support Services (T&MSS) contractor personnel.

### 4.16 Sample Overview Committee (SOC)

The SOC evaluates investigators' requests to examine and acquire samples from the SMF and ensures that samples are available for the complete spectrum of YMP needs and that an appropriate selection of samples is available for posterity. The SOC consists of representatives of Project participants.

The SOC chairman makes recommendations to the Project Office concerning allocation of all samples. The SOC chairman notifies investigators that samples are available for distribution after authorization by the Project Office.

### **5.0 RESPONSIBILITIES**

### 5.1 Principal Investigator

LANL's principal investigator (PI), or designated replacement, is responsible for the identification and control of samples involved in YMP investigations with respect to that PI's study plan. The PI is responsible for submitting sample requests through the SOC representative as described in Section 6.5 and for preparation of detailed technical procedures (DP) that ensure that these samples meet the scientific objectives of the investigations. Samples that are requested from the SMF become the PI's responsibility after they are transferred to him. The PI must also ensure that these requirements, which include all necessary documentation and chain of custody, are met whenever samples are transferred among individuals, groups, or organizations.

### 6.0 **PROCEDURES**

#### 6.1 Introduction

Many of the samples collected for use in Project studies will be the primary source of technical data used in the characterizing Yucca Mountain. It is, therefore, imperative that sample identification and control be sufficient to trace a sample and its derivatives from or to their original field collection location and to document their use in testing or analyses. Traceability includes identification of the chain of custody. Samples needed by the YMP for site characterization activities are catalogued and stored at the SMF.

The identification and control of samples include the following activities: collection, identification, handling, transportation, storage, retrieval, and documentation. General procedures are described in the Project Office administrative procedures (AP) and in the LANL QPs listed in Section 3. Most samples used in site characterization activities will be ultimately stored at the SMF. These samples will be identified and controlled by Project Office branch technical procedures (BTP). However, it is LANL's DPs that ensure that samples meet the technical objectives for which the samples were collected and that describe the documentation necessary to ensure that chain of custody can be established and that an approved quality assurance (QA) plan has been followed.

The major sample categories described herein are (1) bulk samples from the ESF muck pile, (2) core samples from surface-based boreholes, (3) other

samples, which include samples collected from surface sources or from ESF shafts and drifts after mining activities have been completed, and (4) samples sent to and from the SMF. Tables that show the flow of samples in these four categories are included in Attachments 1-4.

# 6.2 Bulk Samples from the ESF Muck Pile

This section identifies how bulk samples from the muck pile are collected during excavation of the ESF. SMF personnel coordinate acquisition of muck from the ESs and drifts. This process includes collection of samples from the ESF and transportation to the SMF, distribution and storage of samples, and the documentation necessary to ensure traceability. Guidelines are described in AP 6.6Q.

### 6.2.1 Collection of Bulk Samples

LANL investigators will not be allowed to collect samples underground during mining activities. SMF personnel will collect bulk samples from the muck pile and transport them to bins at the SMF. Some special sampling needs, such as the protection of samples for  $^{36}$ Cl analysis from any water contamination and the collection of entire muck rounds, are described in AP 6.6Q. Other special sampling needs must be approved by the SOC chairman. If approved, the PI and SOC determine the details of implementation.

The PIs may request that unusual or unexpected features (e.g., a gel) encountered during the mining and mapping of shafts and drifts be collected by miners or other underground personnel. A DP will be prepared that describes how such samples are to be recognized and collected. It is the responsibility of the PI to contact the SMF to ensure that whoever is to collect these samples has an approved procedure for sample collection (acceptable to the PI) or is able to use a DP that describes unusual or unexpected features.

Bulk samples collected during drift mining may not be available for examination but may be loaded directly into 55-gal barrels, transported to, and stored at the SMF. Access to these samples is described in Section 6.5. The PIs may request through the SOC that some drift material, such as material from an interval that includes a fault zone, be deposited in the bins at the SMF so that this material can be examined.

# 6.2.2 Examination of Bulk Samples at the SMF

Muck transferred to the SMF will generally be available for inspection for 7 days, during which time investigators may examine and select the samples they want. All LANL investigators who wish to examine and select samples from the muck must obtain permission to visit the SMF from the Project Office according to AP 6.3Q. The selection of samples is described in the appropriate DPs.

# 6.2.3 Removal and Distribution of Bulk Samples

The removal of bulk samples of muck from the SMF will be authorized by the Project Office upon the recommendation of the SOC. This office will normally grant blanket authorization to PIs to remove shaft and drift material. This approach allows the PI access to all muck samples during the period they are displayed and removes the requirement for specific authorization to be granted to remove individual specimens.

After the PIs have selected material from a bin, SMF personnel distribute the samples and provide documentation and packaging materials. A unique designator in the form of a machine-scanable bar code is assigned to each sample. The sample collection report form (AP 6.3Q) provides documentation. The PI may subsequently remove the sample from the SMF or ship it in accordance with BTP-SMF-006.

After all interested parties have selected the material they need, two 55-gal barrels of archival material will be stored by SMF personnel. These samples can be obtained by following the SMF procedures for requesting samples (Section 6.5).

6.3 Core Samples from Surface-Based Boreholes

Sample-handling activities at YMP borehole sites are described in BTP-SMF-008 and AP 5.2. A drilling program package for each hole will be available at the drill site for the purpose of controlling activities at the drill site. The shift supervisor authorizes any deviations from the drilling program package and resolves issues affecting the collection and documentation of borehole samples. SMF personnel process borehole samples for archival and research needs according to BTP-SMF-004. Processing includes slabbing, photographing, and marking. Requests for core samples or to withhold a specimen from processing must have the SOC chairman's approval.

6.3.1 Collection of Samples from Surface-Based Boreholes at the Drill Site

Generally, LANL investigators will be present at a drilling site only as observers. The SMF field operations staff or Nevada Test Site support contractors are responsible for all collecting, identifying, documenting, packaging, temporarily storing, and transporting samples to the SMF in accordance with BTP-SMF-002 and #003.

Requests that intervals of core be removed directly from the drill site and transported immediately to an investigator's laboratory must be approved by the SOC chairman. Such requests are justified when the time required for SMF processing would jeopardize the analytical integrity of the core sample. However, no core will be removed from the drill site before it has been photographed and marked with a unique sample identification bar code by SMF personnel, as described in BTP-SMF-008.

Before removing core from the drill site, field operations staff and the requestor complete a specimen removal checklist and contract (AP 6.2Q). The core specimen and a photocopy of the contract are released directly to the requestor. Sample curation then becomes the responsibility of the investigator, who follows the sample control procedures in the appropriate LANL DPs. Requests for samples requiring special handling must also be approved by the SOC chairman. Special handling includes the collection of cuttings and water samples for  $^{36}$ Cl analyses. A copy of the authorization document must be made available to the field operations drilling staff before special handling procedures can be initiated. Samples that require special handling are generally controlled by DPs.

6.3.2 Examination of Surface-Based Borehole Samples

LANL investigators who want to examine surface-based borehole samples at the SMF submit a sample examination request form (AP 6.3Q). This request must be approved through the SOC and must be received by the SMF before access can be granted (Section 6.5). Visitors must adhere to any restrictions outlined in BTP-SMF-005.

All unprocessed core will be made available to all YMP participants at a core examination meeting. LANL investigators may examine samples and place temporary markers on the core indicating the intervals to be removed. When more than one investigator requests specimens from the same interval, the matter will be resolved at this meeting by those involved. If conflicts are unresolved, the SOC will assign specimens from the core intervals.

Core can also be examined before the core examination meeting. However, no sample removal requests will be accepted until after the core examination meeting is held. SMF personnel are responsible for displaying, reshelving, and generally controlling the samples, as described in BTP-SMF-005.

# 6.3.3 Removal of Surface-Based Borehole Samples from the SMF

Removal of core and other specimens from the SMF is described in BTP-SMF-006. All LANL investigators who want to remove samples from the SMF must submit a specimen removal request form (AP 6.3Q) and obtain approval from the SOC chairman.

Because previously undetected fractures or features in the core sometimes make it difficult to remove core specimens from the exact interval requested, SOC and SMF personnel will arrange for a buffer zone so that the interval may be adjusted up or down to obtain the specimen. SMF personnel will cut the specimen, as requested, and will provide orientation stripes and top and bottom depth marks. Unique permanent bar code labels will be included.

### 6.4 Other Samples

This section describes the procedures for identification and control of samples intended for use in site characterization but not controlled by AP 6.2Q or AP 6.6Q (i.e., ESF and surface borehole samples). These samples include, but are not limited to, hand and bulk rock, desert varnish, alluvium, soils, fluids, biota, or any other surface samples. Also included are core or wall samples from the shafts and drifts, which are collected after ES mining activities have been completed. Acquisition sites may be the ground surface, surface collectors (e.g., air collectors), streams and creeks, outcrops, drifts, trenches, etc. The general procedures for collecting and controlling samples are described in AP 6.3Q. In addition, the LANL DPs address collection, unique identification of each sample, photographic documentation, control, transportation, handling, chain of custody, storage, retrievability, and documentation.

A LANL investigator obtains a sample collection report form (AP 6.3Q) and unique bar code labels from the SMF for each sample (or feature) to be collected. The sample collection report form accompanies the samples if they are sent to the SMF; if the investigator keeps the samples, he sends a copy of the forms to the SMF as soon as is practicable (i.e., usually within 30 days of collection).

6.4.2 Sample Collection and Identification

The methods for collecting samples must meet the technical objectives of the study plan, must be determined in advance, and must use the appropriate equipment and techniques to obtain the intended samples. Actual collection of samples is documented in Project notebooks or fieldbooks in accordance with QP-03.5 or appropriate DPs. Documentation of sample collection and identification must be sufficient to permit tracing a sample and its processed derivatives to the original field location (e.g., trench location, surface outcrop, wall of shaft).

When possible, samples are identified physically by placing the unique bar code label or other unique identifier directly on the sample or its container. Sample numbers must be traceable to the SMF bar code designation.

6.5 Samples to and from the SMF

Samples collected or generated for use in YMP site characterization studies may be stored at the SMF or at LANL. However, notwithstanding LANL documentation requirements (e.g., QP 17.1), some documentation on all samples from which data are extracted to support the license application will be maintained at the SMF.

All LANL investigators' requests to examine or secure samples from the SMF are coordinated through LANL'S SOC representative in accordance with AP 6.3Q. General use of the SMF is covered in AP 6.3Q and BTP-SMF-001.

6.5.1 Access to SMF

All LANL investigators who wish to visit and secure samples from the SMF must apply for and receive access authorization in writing from the Project Office before visiting the SMF. Access is limited to authorized persons with valid scientific or regulatory needs. Investigators who visit the SMF regularly will be placed on a permanent access list. Those who wish to obtain samples but do not need to visit the SMF can do so by following the procedures in Section 6.5.3.

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In order to examine archived or other samples at the SMF, the investigator must fill out a sample examination request form (AP 6.3Q), submit it to the SOC, and receive permission before visiting the SMF. Examination and acquisition of ESF muck samples are described in Section 6.2. Examination and acquisition of samples from surface-based borehole cores are described in Section 6.3.

Geologic samples collected for the YMP before May 1, 1988, are also available for examination. However, in addition to the above requirements, an investigator must complete an unqualified samples examination agreement form (AP 6.3Q) before examining these samples.

### 6.5.3 Acquisition of Samples

Project Office authorization for removing samples from the SMF are coordinated with the SOC, which may recommend for or against removal authorization, depending on the availability of samples and requests from other participants. LANL investigators who request samples from the SMF complete a specimen removal request (AP 6.3Q). These requests for samples or specimens are normally submitted through LANL's SOC representative. The SOC chairman must approve requests before they are submitted to the SMF.

A specimen removal contract form (AP 6.3Q) is completed to document the transfer of samples from the SMF to the LANL investigator. Each contract contains specifics on the samples or specimens the investigator receives and a list of the tests to be performed. The contract is completed at the SMF if the LANL investigator receives the specimens at the SMF. If the material is shipped to the investigator, he signs and returns the contract to the SMF within 10 working days of receipt of the material.

# 6.5.4 Submission of Samples, Specimens, and Remnants to the SMF

In accordance with AP 6.3Q, each LANL investigator returns all the remaining material from samples, specimens, and remnants at the completion of his research or when the material is no longer needed. Pls should contact the SMF if there is any question as to whether the sample can be stored at the SMF. For example, the SMF will store fluid samples only at the request of Pls. If such samples are to be submitted, it is the responsibility of the PI to prepare the sample (e.g., to acidify the water) for storage. The PI should also state the expected life of the sample.

Investigators submit a completed sample collection report form (AP 6.3Q) for samples, specimens, and remnants not covered by QP 6.2Q. They also affix SMF code labels to the samples or sample containers and to the sample collection report form. An inventory list that identifies the original sample and tests performed on each sample accompanies the material returned. A list of sample identification numbers and their corresponding bar code numbers also accompanies the material, as appropriate.

SMF personnel verify the inventory list and send a copy of the sample collection report, which signifies transfer of custody to the SMF, to the investigator. If any discrepancies are identified, the SMF contacts the investigator and the discrepancies are resolved as specified by BTP-SMF-001.

### 6.6 Sample Curation

LANL investigators assume custody and responsibility for sample curation when they collect or receive samples. The procedures for sample identification, chain-of-custody documentation, and sample marking and labeling are identified in the appropriate DPs.

Samples may be temporarily stored at LANL or subcontractor facilities, provided that the investigator has an approved QA procedure for sample curation and that storage procedures are covered by an appropriate DP.

Samples must be stored under physical conditions appropriate for their intended purpose and expected maximum storage life. Samples intended for long-term storage (as defined by the study plan or PI) are treated in a manner that ensures that they do not degrade during storage. The analytical history and chain of custody of the sample while it is at LANL are documented in a field notebook, laboratory notebook or logbook, or electronic data base.

### 6.7 Shipping

Shipping methods are described in LANL QPs and DPs. Completed shipping forms document the transfer of custody of samples to or from LANL investigators and are filed according to QP 17.1. Container requirements, methods of handling, any environmental or safety considerations, or other items of concern are identified. Where DPs do not exist, QP 13.1 (Procedure for Handling, Storage, and Shipment) will be followed. Controls will be implemented to ensure that sample identification is verified and that documentation is maintained when samples are handled or transferred from one location to another, or from one organization to another, so that chain-of-custody documentation can be maintained.

The PI visually determines whether any samples received at LANL are unacceptable for the purpose for which they were requested. Samples that have been damaged or that have deteriorated are returned to the sender with documentation of how the sample was received and why the sample is being returned. A copy of this information is submitted to the group Resident File (QP 17.1).

If samples deteriorate or become damaged while being used or stored at LANL, the PI determines whether further use of the sample is appropriate and records such information in the appropriate laboratory notebook, logbook, or other sample-tracking system.

### 7.0 QA REQUIREMENTS

#### 7.1 Records

A records package is kept to implement this procedure whenever samples are collected, received, or sent by a LANL investigator. The records package

consists of forms generated during the acquisition and transfer of samples such as the appropriate SMF access and handling forms; shipping manifests identified in AP 6.2Q, AP 6.3Q, AP 6.6Q; various SMF procedures; and any other documents submitted to the group Resident File concerning samples or their condition.

### 8.0 ACCEPTANCE CRITERIA

The criteria that show that this procedure has been correctly implemented are the records identified in Section 7.1, which bear all necessary signatures.

### 9.0 ATTACHMENTS

Attachment 1--Flow of Bulk Samples from the ESF Muck Pile Attachment 2--Flow of Core Samples from Surface-Based Boreholes Attachment 3--Flow of Other Samples Attachment 4--Flow of Samples from the SMF

ltem	Responsible Individual or Organization	Forms and Procedures
Collection and identification of samples (includes issue of bar code label)	SMF	Sample collection report form (AP 6.3Q)
Special sample-handling or collection needs	Pl	Approval of SOC chairman
Examination of samples	PI	YMP permission to visit SMF
Removal and distribution of sample	SMF	Sample collection report form (AP 6.3Q) YMP permission to remove samples, based on SOC recommendation
Sample curation	PI	Approved procedure for curation
Remnant return	Pi	Sample collection report form AP 6.3Q) Inventory list
		Shipping manifest

# FLOW OF BULK SAMPLES FROM THE ESF MUCK PILE

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Responsible Individual or Organization	Forms and Procedures
SMF, NTS	Drilling program package
Pl	SOC approval
	Specimen removal checklist and contract form (AP 6.2Q)
. Pi	SOC-approved authorization document
Pi	YMP approval needed to visit SMF
	Sample examination request form (AP 6.3Q) submitted through SOC
PI	SOC approval, submittal of specimen removal request form (AP 6.3Q) to SMF
SMF	Specimen removal contract form (AP 6.3Q)
	Shipping manifest
PI	Approved procedure for sample curation
PI	Inventory list
	Responsible Individual or Organization SMF, NTS PI PI PI PI SMF PI PI

# FLOW OF CORE SAMPLES FROM SURFACE-BASED BOREHOLES

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# FLOW OF OTHER SAMPLES

ltem	Responsible Individual or Organization	Forms and Procedures
Sample collection forms and SMF bar code labels	PI	Sample collection report forms (AP 6.3Q) and SMF bar code labels
Sample collection and identification	PI.	Appropriate DPs
Shipping	PI	Appropriate DPs Shipping manifest
Curation	PI	Approved procedure for sample curation
Submittal of samples to SMF	PI	Sample collection report form (AP 6.3Q) Inventory list Shipping manifest

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ltem	Responsible Individual or Organization	Forms and Procedures
Access to SMF	PI	Access authorization from YMP
Sample examination	PI	Sample examination request form (AP 6.3Q) to SOC approved and sent to SMF before visit
Sample acquisition	PI	YMP approval needed
		Specimen removal request form (AP 6.3Q) submitted to SMF through SOC
	SMF	Transfer of samples via specimen removal contract form (AP 6.3Q)
Acquisition of unqualified samples	PI	Same as for sample acquisition, plus unqualified samples examination agreement form (AP 6.3Q) submitted to SMF through SOC
Shipping and handling	PI	Appropriate DPs
		Shipping manifests
Submittal of samples and remnants to SMF	PI	Sample collection report form (AP 6.3Q) Inventory list

# FLOW OF SAMPLES TO OR FROM THE SMF

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