Remedial Action Contract
for Remedial Response, Enforcement Oversight, and Non-Time Critical Removal Activities at Sites of Release or Threatened Release of Hazardous Substances in EPA Region VIII

U.S. EPA Contract No. EP-W-05-049

Sampling and Analysis Plan/Quality Assurance Project Plan: Fish and Game Tissue Assessment Libby Asbestos Site, Operable Unit 4

Revision 0 – August 2012

Work Assignment No.: 329-RICO-08BC
Libby Asbestos Superfund Project,
OU4 Remedial Investigation/
Feasibility Study
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A PROJECT MANAGEMENT

A1. Title and Approval Page

Title:

Sampling and Analysis Plan/Quality Assurance Project Plan:
Fish and Game Tissue Assessment
Libby Asbestos Site, Operable Unit 4
Revision 0 – August 2012

Approvals:

Approved by: _____ Date: 9/5/12
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Approved by: _____ Date: 9/5/12
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Approved by: _____ Date: 9/4/12
Elizabeth Fagen
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Approved by: _____ Date: 9/4/12
Victor Ketellapper, P.E.
EPA Region 8, Libby Asbestos Project Team Leader

Sampling and Analysis Plan/Quality Assurance Project Plan: Fish and Game Tissue Assessment Libby Asbestos Site, Operable Unit 4

REVISION LOG:

Revision No.	Date	Description
0	8/31/12	---

A2. Table of Contents

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List of Acronyms and Abbreviations

%	percent
≥	greater than or equal to
µm	micrometers
ASTM	American Society for Testing and Materials
ATV	all-terrain vehicle
CDM Smith	CDM Federal Programs
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHISQ	chi-squared
CI	confidence interval
COC	chain-of-custody
DE	Data Entry
DQO	data quality objective
EDD	electronic data deliverable
EDS	energy dispersive spectroscopy
EDXA	energy dispersive x-ray analysis
EPA	U.S. Environmental Protection Agency
ERT	Environmental Response Team
ESAT	Environmental Service Assistance Team
FSDS	field sample data sheet
FTL	field team leader
GIS	geographic information system
GPS	global positioning system
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
ID	identification
IDW	investigation-derived waste
ISO	International Organization for Standardization
L^{-1}	per liter
LA	Libby amphibole
LADT	Libby Asbestos Data Tool
LC	laboratory coordinator
MDEQ	Montana Department of Environmental Quality
MFWP	Montana Fish, Wildlife and Parks
mm^2	square millimeters
N	number
NFG	National Functional Guidelines
NIST	National Institute of Standards and Technology
NTP	National Toxicology Program
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration
OU	operable unit

PAPR	positive air pressure respirator
PPE	Personal protective equipment
QA	quality assurance
QA/QC	quality assurance/quality control
QAM	Quality Assurance Manager
QAPP	quality assurance project plan
QATS	Quality Assurance Technical Support
QC	quality control
ROM	record of modification
RPM	Regional Project Manager
s/g	structures per gram
SAED	selective area electron diffraction
SAP	sampling and analysis plan
Shaw	Shaw Environmental, Inc.
Site	Libby Asbestos Superfund Site
SOP	standard operating procedure
SRM	standard reference material
TEM	transmission electron microscopy
USGS	United States Geological Survey

A3. Distribution List

Copies of this completed and signed sampling and analysis plan/quality assurance project plan (SAP/QAPP) should be distributed to:

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Denver, Colorado 80202-1129

- Victor Ketellapper, Ketellapper.Victor@epa.gov (1 hard copy, electronic copy)
- Elizabeth Fagen, Fagen.Elizabeth@epa.gov (electronic copy)
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- Damon Repine, repineDL@cdmsmith.com (electronic copy)

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Copies of the SAP/QAPP will be distributed to the individuals above by CDM Federal Programs (CDM Smith), either in hard copy or in electronic format (as indicated above). The CDM Smith Project Manager (or their designee) will distribute updated copies each time a SAP/QAPP revision occurs. An electronic copy of the final, signed SAP/QAPP (and any subsequent revisions) will also be posted to the Libby Field eRoom[1].

A4. Project Task Organization

Figure A-1 presents an organizational chart that shows lines of authority and reporting responsibilities for this project. The following sections summarize the entities and individuals that will be responsible for providing project management, technical support, and quality assurance for this project.

A4.1 Project Management

The U.S. Environmental Protection Agency (EPA) is the lead regulatory agency for Superfund activities within the Libby Asbestos Superfund Site (Site). The EPA Region 8 Libby Asbestos Project Team Leader is Victor Ketellapper. The EPA Regional Project Manager (RPM) for this sampling effort is Elizabeth Fagen. The EPA Region 8 Onsite Field Team Leader (FTL) for this sampling effort is Michael Cirian.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities at the Site. The MDEQ Project Manager for this sampling effort is Carolyn Rutland. The EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities.

A4.2 Technical Support

A4.2.1 SAP/QAPP Development

This SAP/QAPP was developed by CDM Smith at the direction of, and with oversight by, the EPA. This SAP/QAPP contains all the elements required for both a SAP and a QAPP and has been developed in general accordance with the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA 2001) and the *Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4* (EPA 2006).

Copies of the SAP/QAPP will be distributed by the CDM Smith Project Manager (or their designee), either in hard copy or in electronic format, as indicated in Section A3.

[1] https://team.cdm.com/eRoom/R8-RAC/Libby

A4.2.2 Field Sampling Activities

Fish have already been collected by Golder Associates, Inc. (a subcontractor to Remedium Group, Inc.) as part of the Phase V Part B sampling program (EPA 2012a) for Operable Unit 3 (OU3).

EPA Region 8 staff will be responsible for collecting game tissue samples. CDM Smith will provide logistical support (e.g., completing field documentation, sample packaging and shipping) for field sampling activities for game as described in this SAP/QAPP. CDM Smith personnel that will be involved in this sampling program include:

- Nathan Smith, Project Manager
- Tracy Dodge, Sample Coordinator
- Scott Miller, Field Data Manager
- Terry Crowell, Quality Assurance Manager
- Damon Repine, Health and Safety Manager

EPA personnel that will be involved in the field sampling of game and collection of tissues include:

- Elizabeth Fagan, Field Supervisor
- Michael Cirian, Onsite Field Team Leader
- Deborah McKean, Toxicologist
- David Berry, Toxicologist

A4.2.3 Asbestos Analysis

All samples collected as part of this project will be sent for preparation and analysis for asbestos at EMSL Analytical, Inc. in Cinnaminson, New Jersey (see Section B4). The EPA Environmental Services Assistance Team (ESAT) is responsible for procuring all analytical laboratory services and providing direction to the analytical laboratories. Don Goodrich (EPA Region 8) is responsible for managing the ESAT laboratory support contract for asbestos. The ESAT Region 8 Team Manager at TechLaw, Inc. is Mark McDaniel. He is also the designated laboratory coordinator (LC) for the Libby project that is responsible for directing the analytical laboratories, prioritizing analysis needs, and managing laboratory capacity.

A4.2.4 Data Management

All data generated as part of this sampling effort will be managed and maintained in Scribe (see Section B10). The EPA Environmental Response Team (ERT) is responsible for the administration of all Scribe data management aspects of this project. Joseph Schafer is responsible for overseeing the ERT data management support contract. ERT is responsible for

the development and management of Scribe and the project-specific data reporting requirements for the Libby project.

The CDM Smith field data manager (Scott Miller) is responsible for uploading sample information to the field Scribe project database. ESAT is responsible for uploading new analytical results to the analytical Scribe project database. The ESAT project data manager for the Libby project is Janelle Lohman (TechLaw, Inc.).

Because of the quantity and complexity of the data collected at the Site, the EPA has designated a Libby Data Manager to manage and oversee the various data support contractors. The EPA Region 8 Data Manager for the Libby project is Jeff Mosal.

A4.3 Quality Assurance

There is no individual designated as the EPA Quality Assurance Manager (QAM) for the Libby project. Rather, the Region 8 quality assurance (QA) program has delegated authority to the EPA RPMs. This means that the EPA RPMs have the ability to review and approve governing investigation documents developed by Site contractors. Thus, it is the responsibility of the EPA RPM for this sampling effort (Elizabeth Fagen), who is independent of the entities planning and obtaining the data, to ensure that this SAP/QAPP has been prepared in accordance with the EPA QA guidelines and requirements. The EPA RPM is also responsible for managing and overseeing all aspects of the quality assurance/quality control (QA/QC) program for this sampling effort. In this regard, the RPM is supported by the EPA Quality Assurance Technical Support (QATS) contractor, Shaw Environmental, Inc. (Shaw). The QATS contractor will evaluate and monitor QA/QC sampling and is responsible for performing annual audits of each analytical laboratory.

Terry Crowell (CDM Smith) is the field QAM for this project. Ms. Crowell is responsible for evaluating and monitoring field QA/QC and providing oversight of field sampling and data collection activities.

A5. Problem Definition/Background

A5.1 Site Background

Libby is a community in northwestern Montana located 7 miles southwest of a vermiculite mine that operated from the 1920s until 1990. The mine began limited operations in the 1920s and was operated on a larger scale by the W.R. Grace Company from approximately 1963 to 1990. Studies revealed that the vermiculite from the mine contains amphibole-type asbestos, referred to as Libby amphibole (LA).

Epidemiological studies revealed that workers at the mine had an increased risk of developing asbestos-related lung disease (McDonald *et al.* 1986, Amandus and Wheeler 1987, Sullivan 2007). Additionally, radiographic abnormalities were observed in 17.8 percent of the general population of Libby, including former workers, family members of workers, and individuals with no specific pathway of exposure (Peipins *et al.* 2003). Although the mine has ceased operations, historic or continuing releases of LA from mine-related materials could be serving as a source of on-going exposure and risk to current and future residents and workers in the area. The Site was listed on the National Priorities List in October 2002.

A5.2 Reasons for this Project

Historic mining and milling operations at the Site have resulted in the release of LA to the environment. Previous investigations have demonstrated that LA is present in soils, sediments, surface water, soil, duff[2], and tree bark at the Site, with concentrations tending to be highest on the mine site and the forested areas immediately surrounding the mine (i.e., OU3). Although the exposure pathway of primary concern for humans is inhalation of LA, some studies in animals suggest that ingestion of asbestos fibers can result in the growth of benign intestinal polyps (National Toxicology Program [NTP] 1985). There are several potential scenarios where humans could be exposed to LA *via* ingestion. Two exposure scenarios are as follows:

Ingestion of Game. One exposure scenario is the ingestion of edible tissue from hunted game that forage in OU3. Game, including deer and elk, have been observed within OU3 and may be exposed to LA in a variety of potential exposure media. It is possible that these animals may have accumulated LA in their tissues as a result of these exposures. However, at present, there are no data available on the potential concentrations of LA in edible game tissue at the Site. Thus, data are needed that can be used to determine if human exposures from ingestion of LA in game tissue have the potential to cause unacceptable health risks.

Ingestion of Fish. Another exposure scenario is the ingestion of LA in edible fish tissue (fillets) from fish caught from local streams and ponds that contain LA. However, at present, there are no data available on the potential concentrations of LA in edible fish tissue at the Site. Thus, data are needed that can be used to determine if human exposures to LA from ingestion of edible fish tissue have the potential to cause unacceptable health risks. As noted above, the collection of fish is included as part of the OU3 Phase V Part B study (EPA 2012a); but the sample preparation (filleting) and analysis of these fish is being conducted under this SAP/QAPP.

[2] Duff is comprised of un-decomposed twigs, needles, and other vegetation and the layer of partially- to fully-decomposed litter that occurs on top of the mineral soil in forested areas.

A5.3 Applicable Criteria and Action Limits

There are no LA-specific criteria or action limits for tissue burdens that apply to this sampling program.

A6. Project/Task Description

A6.1 Task Summary

Basic tasks that are required to implement this SAP/QAPP include collecting two game animals (deer and elk) from OU3, collecting tissue samples (i.e., muscle, heart, liver, lung, kidney, and diaphragm) from these game animals, preparing fillets for fish collected from OU3, and analyzing asbestos in game tissues and fish fillets. These basic tasks are described in greater detail in subsequent sections of this SAP/QAPP.

A6.2 Work Schedule

Fish were collected from OU3 in August of 2012 (EPA 2012a). Game tissue will be collected in the fall of 2012. Sample analysis and data evaluation and interpretation tasks will be performed over the winter of 2012/2013.

A6.3 Locations to be Evaluated

Game animals will be collected from OU3 in areas surrounding the mine site (see **Figure A-2**) where deer and elk are known to forage. The specific area for collection of the game animals will depend upon the availability and location of the game animals. Fish were collected from the Mill Pond located in OU3 (see **Figure A-3**).

A6.4 Resources and Time Constraints

As noted above, the fish collection occurred in August 2012. The intent is to collect one deer and one elk during cooler weather (fall 2012) when it will be easier to transport the animals from the mine site to the processing area and to perform dissections to obtain tissues. It is also necessary to collect elk prior to their movement to lower elevations in late fall.

A7. Quality Objectives and Criteria

A7.1 Data Quality Objectives

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. The design of a study is closely tied to the DQOs, which serve as the basis for important decisions regarding key design features such as the number and location

of samples to be collected and types of analyses to be performed. The EPA has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific decision-making (EPA 2001; 2006).

Appendix A provides the detailed implementation of the seven-step DQO process associated with the collection of game tissues. The DQOs for the collection of fish tissues are included in the *SAP/QAPP, OU3, Libby Asbestos Superfund Site, Phase V Part B: 2012 Ecological Studies* (EPA 2012a).

A7.2 Performance Criteria

The expected range of LA concentrations in game and fish tissues is not known. However, it is possible to estimate the concentration levels that correspond to a level of human health concern. The analytical requirements for LA measurements established in Section B4 ensure that tissue burden results from this study will provide data that are adequate to support risk management decision-making.

Section D3 provides additional information on how data users should perform a data usability assessment to ensure that data from this study are adequate with respect to the DQOs.

A7.3 Precision

The precision of asbestos measurements is determined mainly by the number (N) of asbestos structures counted in each sample. The coefficient of variation resulting from random Poisson counting error is equal to $1/N^{0.5}$. In general, when good precision is needed, it is desirable to count a minimum of 3-10 structures per sample, with counts of 20-25 structures per sample being optimal.

Field duplicates of each type of tissue sample will be collected (see Section B5.1.5). In addition, three filter replicates of each tissue sample will be analyzed (see Section B4.1.1). Analysis of these field duplicates and filter replicates will provide a measure of the precision of the sampling and analysis process. Recount and repreparation analyses will also be performed (see Section B5.2.4) to provide information on analysis reproducibility and precision.

A7.4 Bias/Accuracy and Representativeness

There is no established set of reference materials or spiked standards that can be used to assess the accuracy of transmission electron microscopy (TEM) analyses of LA in tissues. But, a review of results for field blanks (see Section B5.1.5) and laboratory blanks (see Section B5.2.4) will ensure that results are not biased due to cross-contamination, either in the field or the analytical laboratory.

Game animals and fish will be collected from an area where exposures are expected to be highest (i.e., OU3). Thus, resulting tissue concentrations could result in exposures that are potentially biased high. For game animals, since the amount of time that the animals may have foraged within OU3 is not known, the magnitude of the potential bias is not known for game. For fish, because migration of fish to/from the Mill Pond is unlikely[3], and measured concentrations in water and sediment from this pond are higher than areas that are likely to be frequented by anglers (e.g., Kootenai River), tissue burdens for fish from the Mill Pond are likely to represent the high-end of potential human exposures.

A7.5 Completeness

Target completeness for this project is 100 percent (%). If any samples are not collected, or if LA analysis is not completed successfully, this could result in that portion of the study providing no useful information. In this event, additional sampling may be needed to support EPA decision-making.

A7.6 Comparability

Although animal tissue samples for the purposes of establishing LA tissue burdens have not been collected previously, the data generated during this study will be obtained using sample preparation and analysis methods for measuring LA that have been established for the Libby Site for other media (e.g., duff, tree bark).

A7.7 Method Sensitivity

The method sensitivity (analytical sensitivity) needed for LA analysis of tissue is discussed in Section B4.

A8. Special Training/Certifications

A8.1 Field

Asbestos is a hazardous substance that can increase the risk of cancer and serious non-cancer effects in people who are exposed by inhalation. Therefore, all individuals involved in the collection of samples must have appropriate training. Prior to starting any field work, field team members must have the following special training and certifications:

[3] Lower Rainy Creek (where the Mill Pond is located) is isolated from upward migration of fish from the Kootenai River by a hanging culvert and from downward migration of fish from Upper Rainy Creek by the tailings impoundment.

- Read and understand the governing Health and Safety Plan (HASP),
- Occupational Safety and Health Administration (OSHA) 40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) and relevant 8-hour refreshers
- Current 40-hour HAZWOPER medical clearance
- Respiratory protection training as required by 29 CFR 1910.134
- Asbestos awareness training, as required by 29 CFR 1910.1001

Accordingly, in addition to other health and safety procedures established in the governing HASP, all field personnel working on OU3 will wear protective garments and respiratory protection appropriate to HAZWOPER protection Level C. Respiratory protection will be *via* positive air pressure respirators (PAPRs).

Prior to beginning field sampling activities, a field planning meeting will be conducted to discuss and clarify the following:

- Objectives and scope of the fieldwork
- Equipment and training needs
- Field operating procedures, schedules of events, and individual assignments
- Required quality control (QC) measures
- Health and safety requirements

It is the responsibility of each field team member to review and understand all applicable governing documents associated with this sampling program, including this SAP/QAPP, all associated standard operating procedures (SOPs) (see **Appendix B**), and the governing HASP.

A8.2 Laboratory

A8.2.1 Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Each laboratory is accredited by the National Institute of Standards and Technology (NIST)/National Voluntary Laboratory Accreditation Program (NVLAP) for the analysis of airborne asbestos by TEM. This includes the analysis of NIST/NVLAP standard reference materials (SRMs), or other verified quantitative standards, and successful participation in two proficiency rounds per year of airborne asbestos by TEM supplied by NIST/NVLAP.

Copies of recent proficiency examinations from NVLAP or an equivalent program are maintained by each participating analytical laboratory. Many of the laboratories also maintain certifications from other state and local agencies. Copies of all proficiency examinations and certifications are also maintained by the LC.

Each laboratory working on the Libby project is also required to pass an on-site EPA laboratory audit. The details of this EPA audit are discussed in Section B5.2.3. The LC also reserves the right to conduct any additional investigations deemed necessary to determine the ability of each laboratory to perform the work. Each laboratory also maintains appropriate certifications from the state and possibly other certifying bodies for methods and parameters that may also be of interest to the Libby project. These certifications require that each laboratory has all applicable state licenses and employs only qualified personnel. Laboratory personnel working on the Libby project are reviewed for requisite experience and technical competence to perform asbestos analyses. Copies of personnel resumes are maintained for each participating laboratory by the LC in the Libby project file.

A8.2.2 Laboratory Team Training/Mentoring Program

Initial Mentoring

The orientation program to help new laboratories gain the skills needed to perform reliable analyses at the Site involves successful completion of a training/mentoring program that was developed for new laboratories prior to their analysis of Libby field samples. All new laboratories are required to participate in this program. The training program includes a rigorous 2-3 day period of on-site training provided by senior personnel from those laboratories already under contract on the Libby project, with oversight by the QATS contractor. The tutorial process includes a review of morphological, optical, chemical, and electron diffraction characteristics of LA, as well as training on project-specific analytical methodology, documentation, and administrative procedures used on the Libby site. The mentor will also review the analysis of at least one sample by each type of analytical method with the trainee laboratory.

Site-Specific Reference Materials

Because LA is not a common form of asbestos, U.S. Geological Survey (USGS) prepared Site-specific reference materials using LA collected at the Libby mine site (EPA 2008a). Upon entry into the Libby program, each laboratory is provided samples of these LA reference materials. Each laboratory is required to analyze multiple LA structures present in these samples by TEM in order to become familiar with the physical and chemical appearance of LA and to establish a reference library of LA energy dispersive spectroscopy (EDS) spectra. These laboratory-specific and instrument-specific LA reference spectra (EPA 2008b) serve to guide the classification of asbestos structures observed in Libby field samples during TEM analysis.

Regular Technical Discussions

On-going training and communication is an essential component of QA for the Libby project. To ensure that all laboratories are aware of any technical or procedural issues that may arise, a

regular teleconference is held between the EPA, their contractors, and each of the participating laboratories. Other experts (e.g., USGS) are invited to participate when needed. These calls cover all aspects of the analytical process, including sample flow, information processing, technical issues, analytical method procedures and development, documentation issues, project-specific laboratory modifications, and pertinent asbestos publications.

Professional/Technical Meetings

Another important aspect of laboratory team training has been the participation in technical conferences. The first of these technical conferences was hosted by USGS in Denver, Colorado, in February 2001, and was followed by another held in December 2002. The Libby laboratory team has also convened on multiple occasions at the American Society of Testing and Materials (ASTM) Johnston Conference in Burlington, Vermont, including in July 2002, July 2005, July 2008, and July 2011, and at the Michael E. Beard Asbestos Conference in San Antonio, Texas in January 2010. In addition, members of the Libby laboratory team attended an EPA workshop to develop a method to determine whether LA is present in a sample of vermiculite attic insulation held in February 2004 in Alexandria, Virginia. These conferences enable the Libby laboratory and technical team members to have an on-going exchange of information regarding all analytical and technical aspects of the project, including the benefits of learning about developments by others.

A8.2.3 Analyst Training

All TEM analysts for the Libby project undergo extensive training to understand TEM theory and the application of standard laboratory procedures and methodologies. The training is typically performed by a combination of personnel, including the laboratory manager, the laboratory QAM, and senior TEM analysts.

In addition to the standard TEM training requirements, trainees involved with the Libby project must familiarize themselves with Site-specific method deviations, project-specific documents, and visual references. Standard samples that are often used during TEM training include known pure (traceable) samples of chrysotile, amosite, crocidolite, tremolite, actinolite and anthophyllite, as well as fibrous non-asbestos minerals such as vermiculite, gypsum, antigorite, kaolinite, and sepiolite. New TEM analysts on the Libby project are also required to perform an EDS spectra characterization study (similar to EPA 2008b) on the LA-specific reference materials provided during the initial training program to aide in LA mineralogy recognition and definition. Satisfactory completion of each of these tasks must be approved by a senior TEM analyst.

All TEM analysts are also trained in the Site-specific laboratory QA/QC program requirements for TEM (see Section B5.2.4). The entire program is discussed to ensure understanding of requirements and responsibilities. In addition, analysts are trained in the project-specific reporting requirements and data reporting tools utilized in transmitting results. Upon

completion of training, the TEM analyst is enrolled as an active participant in the Libby laboratory program.

A training checklist or logbook is used to assure that the analyst has satisfactorily completed each specific training requirement. It is the responsibility of the laboratory QAM to ensure that all TEM analysts have completed the required training requirements.

A9. Documentation and Records

A9.1 Field

Field teams will record sample information on the most current version of the field sample data sheets (FSDSs) developed for tissue collection[4]. Section B3.1.2 provides detailed information on the documentation requirements for FSDS forms. In brief, the FSDS forms document the unique sample identifier assigned to every tissue sample collected as part of this program. In addition, the FSDSs provide information on whether the sample is representative of a field sample or a field-based QC sample (e.g., field duplicate).

A9.2 Laboratory

All preparation and analytical data for asbestos generated in the laboratory will be documented on Site-specific laboratory bench sheets and entered into a database or spreadsheet electronic data deliverable (EDD) for submittal to the data managers. Section B4.2 provides detailed information on the requirements for laboratory documentation and records.

A9.3 Logbooks and Records of Modification/Deviations

It is the also responsibility of the field team, preparation laboratory, and analytical laboratory staff to maintain logbooks and other internal records throughout the sample lifespan as a record of sample handling procedures. Significant deviations (i.e., those that impact or have the potential to impact investigation objectives) from this SAP/QAPP, or any procedures referenced herein governing sample handling, will be discussed with the EPA Project Manager (or their designate) prior to implementation. Such deviations will be recorded on a Record of Modification (ROM) form. Sections B5.1.2 and B5.2.2 provide detailed information on the procedures for preparing and submitting ROMs by field and analytical laboratory personnel, respectively.

[4] The most recent versions of the FSDS forms are provided in the Libby Field eRoom.

B Data Generation and Acquisition

B1. Study Design

B1.1 Location

The level of LA in game tissue is expected to be related to the concentration of LA in various exposure media (e.g., soil, forest duff, sediment, surface water) and the length of time the game have been exposed (i.e., older game animals are likely to have higher tissue concentrations than younger animals). Thus, to ensure that the evaluation focuses on the high-end of the potential range of exposures, game animals should be collected from a location with high LA concentrations. Studies conducted at OU3 have shown elevated concentrations of LA in various media at the mine site and in the forested areas surrounding the mine site. Thus, the goal is to collect animals that forage in these areas.

B1.2 Sampling Design

The following section provides an overview of the sampling effort for game that will be conducted. Detailed information on sampling procedures and methods are presented in Section B2.

Two game species (deer and elk), one animal for each, will be collected from area(s) within the OU3 site. If an elk cannot be collected within a two-week baiting period, then two deer may be collected. Muscle (flank and backstrap) tissue samples will be collected from each game animal and analyzed for LA. Measured LA concentrations in muscle tissue will be used to determine if human exposures to LA from ingestion of game have the potential to cause unacceptable health risks. Five other types of tissue will be also collected from the game animals, including liver, kidney, lung, diaphragm, and heart, to provide additional information on tissue burdens in game animals.

Fish (trout) were collected from the Mill Pond as part of the implementation of the OU3 Phase V Part B SAP/QAPP (EPA 2012a). These fish are currently frozen in archive at the CDM Smith field office in Libby. These fish will be sent to the analytical laboratory, filleted, and the fillet samples will be analyzed for LA. Measured LA concentrations in fillet tissue will be used to determine if human exposures to LA from ingestion of fish have the potential to cause unacceptable health risks.

B1.3 Study Variables

Based on the expectation that the concentration of LA in tissue will tend to increase over time as a function of the long-term cumulative exposure pattern, it is unlikely that tissue burdens will vary substantially due to short-term fluctuations in media concentrations. Thus, the timing of

the animal collection is primarily based on ease of sample collection and permit requirements. Collection of fish occurred in August 2012 and collection of game animals will likely occur in the fall of 2012.

B1.4 Critical Measurements

The critical measurement associated with this project is the measurement of the concentration of LA in tissue samples from fish, deer, and elk collected from OU3. The analysis of LA may be achieved using several different types of microscope, but the EPA generally recommends using TEM because this technique has the ability to clearly distinguish asbestos from non-asbestos structures, and to classify different types of asbestos (i.e., LA, chrysotile). In addition, the results of the TEM analysis include information on the dimensions and attributes of each asbestos structure observed. This is important for the purposes of comparison to ingestion toxicity values because these values are based on structures longer than 10 micrometers (µm) in length.

In addition, data are needed on the moisture content of the tissues prior to TEM analysis. This will allow for concentrations to be translated from a dry weight basis (the analysis output units) to wet weight basis (the units necessary for evaluating human ingestion exposures).

B1.5 Data Reduction and Interpretation

Data collected from this study will be used to support a risk analysis to determine if human exposures to LA from ingestion of fish and game tissue have the potential to cause unacceptable health risks. The data will be used to estimate the high-end intake and risk to people from ingestion of LA-contaminated fish and game.

B2. Sampling Methods

B2.1 Sample Collection

The following subsections provide specific requirements for sample collection. A list of general field equipment that will be used to perform this sampling is provided in Section B8.1 of this SAP/QAPP.

B2.1.1 Game Animals and Tissues

<u>Target Species</u>

Two game animals are identified for collection, including deer (either mule deer, *Odocoileus hemionus*, or white-tailed deer, *Odocoileus virginianus*) and elk (*Cervus elaphus*). These animals are the most common large game animals consumed by humans in the Libby area. The sampling design for collection of the game animals as presented in this SAP/QAPP meet the

requirements of the Montana Fish, Wildlife and Parks (MFWP) Scientific Collection Permit that will govern the collection of the game animals (see **Appendix C**).

Baiting and Camera Surveillance

In order to identify a location where deer and elk may occur and be collected, selected areas within OU3 will be baited to attract the game animals. Motion activated game cameras will be used to monitor use of the area remotely. The cameras will be periodically visited and photos downloaded. Once use of the area is established by deer and/or elk, the sampler will enter the site and collect the target species. It is possible that the deer and elk will be collected from different locations. Baiting attractants may include grains, corn, and commercially available product to entice the animals to frequent the targeted location. Once animals are harvested, or if bear activity is detected, all baiting materials will be collected and removed to eliminate the temptation for animals to return to area.

Collection of the Target Species

Mike Cirian (EPA) will collect the game animals and is a well-experienced hunter with over 40 years of hunting big game by rifle, muzzle loader, and archery, and has harvested over 50 big game animals (deer, bear, caribou, wild boar, and javelin). He is has Hunting Safety Certificates from both Nebraska (firearm and archery) and Wisconsin (firearm). He is a Shotgun Coach (L1) with Scholastic Clay Target Program, National Rifle Association, a member of the Amateur Trapshooting Association, a member of The United States Practical Shooting Association, a range officer at the Libby Rod and Gun Club, and a member of the Cabinet Rifle and Pistol Association.

Preference will be given to the collection of one female deer[5] and one female elk as the numbers of female animals are expected to be greater compared to males and body size is likely to be smaller and more manageable to transport out of OU3. The game animals will be collected by rifle. To avoid compromising organ tissue samples, head or neck shots will be preferred. If the animals are not killed with the first shot, they will be tracked until taken.

Once harvested, the animals will be removed from the OU3 sampling location to an area that is outside of OU3 for processing. All bait will be cleaned up and removed. The animals will be placed on a tarp and removed from the collection area by use of an all-terrain vehicle (ATV). If necessary due to size, the animal(s) will be quartered and then placed on the tarp before being transported from OU3. The animals will be removed to the concrete pad near the amphitheatre. The game animals will be decontaminated by rinsing with water and dressed and wrapped in new tarps. After decontamination, the animals will be moved to an area outside of OU3 for the

[5] For mule deer, a buck will be preferred in accordance with MFWP hunting regulations (MFWP 2012) for the Libby Valley.

collection of tissue samples. This area will be in a private and secure area that will limit potential cross-contamination and exposures for non-sampling personnel.

Animal Aging

It is expected that the concentration of LA in tissue will tend to increase over time as a function of the long-term cumulative exposure pattern (age). Thus, the preference is to collect animals that are older. In general, body size and antler size can help indicate the age of the deer or elk, but these may be misleading (Jensen 1999). Body size and antler size do not necessarily indicate age, but rather reflect nutritional status and genetics (Foresman 2001).

Tooth eruption and wear in deer are closely related to the age of the animal. Deer shed and replace milk teeth with permanent teeth at consistent ages. A few indicators may help with aging, such as physical shape of animals, "roman" nose and sway back in deer, but these are not guaranties to age of animal.

Elk are aged by tooth development and wear. Elk replace their "baby teeth" with permanent teeth at a relatively set rate. At 1½ years old, an elk will have its central two permanent incisors. By 3½years, all permanent teeth are in. At this stage, estimating age is based largely on the rate of tooth wear. Diet and soil types may accelerate tooth wear, but generally, estimating the age is straight-forward through age 3½ years. In animals 4½ years and older, estimating age by tooth wear is less reliable (Jensen 1999).

As it is important to understand the age of the animals in relation to the tissue burdens, the heads with jawbones and teeth will be collected and taken to local MFWP personnel for aging based on examination after the collection of the target tissues.

Tissue Sample Collection

The game animals will be taken to the tissue collection area and samples will be collected by gross dissections. The gross necropsy and collection of tissue samples will be conducted in accordance with the procedures specified in SOP EPA-LIBBY-2012-15, *Gross Necropsy and Tissue Sample Collection for Game Animals* (see **Appendix B**).

For assessing potential human health risks associated with the ingestion of LA in deer or elk tissue, flank and backstrap muscle samples will be collected. To confirm exposures to asbestos, other target tissues will also be collected, including the heart, liver, lung, kidney, and diaphragm. For each organ type collected, the organ will be split in half and three subsamples collected from one of the halves. The three subsamples will be rinsed and then placed into a single sample container as one sample. Samples will be put on wet ice and shipped immediately to the analytical laboratory.

The remaining organ halves and muscle tissue will be frozen and stored for possible later use. Per the permit (see **Appendix C**), if the remaining meat is deemed to be fit for human consumption (based on the analytical results), it will be donated to the local food bank. The remaining carcass will be disposed of as investigation-derived waste (IDW), as described in Section B.2.4 or left at the harvest site.

B2.1.2 Fish Fillet Tissues

Whole body fish will be filleted by the analytical laboratory in accordance with the procedures specified in SOP EPA-LIBBY-2012-14, *Filleting Fish Samples* (see **Appendix B**). One fillet from each fish will be collected. The remainder of the fish (i.e., the other fillet, organs, carcass) will be frozen and stored for possible later use.

B2.2 Global Positioning System Coordinate Collection

For this investigation, global positioning system (GPS) coordinates will be collected for all bait and camera locations, as well as the specific area where deer and elk are collected. GPS location coordinates will be recorded in basic accordance with Site-specific SOP CDM-LIBBY-09, *GPS Coordinate Collection and Handling* (see **Appendix B**).

GPS coordinates will be collected as Sample Points, requiring the input of sample identification (ID) number (also referred to as index ID) and location ID. Since multiple samples may be attributed to one area, for this sampling program the index ID will be input as 'N/A'.

Field-collected GPS data are converted to a usable geographic information system (GIS) format using the general processes described in SOP CDM-LIBBY-09. After the conversion from GPS points to GIS files, 100% of the data is checked visually to identify any potential data entry errors.

B2.3 Equipment Decontamination

Equipment used to collect, handle, or measure environmental samples will be decontaminated in basic accordance with Site-specific SOP EPA-LIBBY-2012-04, *Field Equipment Decontamination at Nonradioactive Sites* (see **Appendix B**). Materials used in the decontamination process will be disposed of as IDW as described below. This SOP specifies the minimum procedural requirements for equipment decontamination. Additional equipment decontamination procedures are also specified in the medium-specific collection SOPs.

B2.4 Handling Investigation-derived Waste

Any disposable equipment or other IDW will be handled in general conformance with Site-specific SOP EPA-LIBBY-2012-05, *Guide to Handling of Investigation-Derived Waste* (see **Appendix B**). In brief, IDW will be double bagged in clear 6-mil poly bags with 'IDW' written, in letters at

least 3-inches high, in indelible ink on at least two sides of the outer bag. All IDW generated during this sampling program will enter the waste stream at the local class IV asbestos landfill.

B3. Sample Handling and Custody

B3.1 Sample Identification and Documentation

B3.1.1 Field Sample Labels

Tissue samples will be labeled with sample ID numbers. The labels will be affixed to the inside of both the inner and outer sample bags and the sample ID number will be written in indelible ink on the outside of each bag.

Sample ID numbers will identify the samples collected during this sampling effort using the following format:

TS-#####

where:

TS = Prefix that designates samples collected under this **T**issue **S**tudy SAP/QAPP
= A sequential five-digit number

B3.1.2 Field Sample Data Sheets

As noted previously in Section A9, field teams will record sample information on the most current version of the game tissue collection FSDS. Use of standardized forms ensures consistent documentation across samplers. Hard copy FSDSs are location-specific and allow for the entry of up to three individual samples from the same location on the same FSDS form. If columns are left incomplete due to fewer than three samples being recorded on a sheet, the blank columns will be crossed out, dated, and signed by the field team member completing the FSDS. Erroneous information recorded on a hard copy FSDS will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

FSDS information will be completed in the field before field personnel leave the tissue sampling location. To ensure that all applicable data is accurately entered and all fields are complete, a different field team member will check each FSDS. The team member completing the hard copy form and the team member checking the form will initial the FSDS in the proper fields. In addition, the FTL will also complete periodic checks of FSDSs prior to relinquishment of the samples to the field sample coordinator. Once FSDSs and samples are relinquished to the field

sample coordination staff, the FSDSs are again checked for accuracy and completeness when data are input into the local Scribe field database.

If a revision is required to the hard copy FSDS during any of these checks, it will be returned to the field team member initially responsible for its completion. The error will be explained to the team member and the FSDS corrected. If the team member is no longer on site, revisions will be made by sample coordination staff or the FTL. It is the responsibility of the field data manager to make the appropriate change in the local Scribe field database.

Each hard copy FSDS is assigned a unique sequential number. This number will be referenced in the field logbook entries related to samples recorded on individual sheets. Field administrative staff will manage the hard copy FSDSs in their respective field office. Original FSDSs will be filed by medium and FSDS number. Hard copies of all FSDS forms will also be sent to the CDM Smith office in Denver, Colorado for archive.

B3.1.3 Field Logbooks

The field logbook is an accounting of activities at the Site and will duly note problems or deviations from the governing documents. Field logbooks will be maintained in general conformance with Site-specific SOP EPA-LIBBY-2012-01, *Field Logbook Content and Control* (see **Appendix B**).

The cover of the field logbook will clearly indicate the name of the investigation. Field logbooks will be completed for each investigation activity prior to leaving a sampling location. Field logbooks will be checked for completeness and adherence to SOP requirements by the FTL. When incorrect field logbook completion procedures are discovered during these checks, the errors will be discussed with the author of the entry and corrected. Erroneous information recorded in a field logbook will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

As the field logbook is completed, originals will be catalogued and maintained by the field administrative staff in their respective field office. Scanned copies of field logbooks will be maintained on the local servers for the CDM Smith offices in Libby and Denver.

B3.1.4 Photographs and Video

Photographic documentation will be collected with a digital camera in general conformance to SOP EPA-LIBBY-2012-02, *Photographic Documentation of Field Activities* (see **Appendix B**). Photographs should be taken to document the specimen collection and processing, and at any other special conditions or circumstances that arise during the sampling activity.

Electronic captions will be used to describe the photographs instead of maintaining photographic logs in daily logbook entries.

Photograph file names will be in the format:

TS_Date_Desc_##

where:

TS indicates **T**issue **S**tudy

Date is formatted as MM-DD-YY

Desc indicates a general description of the photograph content

indicates a two-digit sequential number

B3.2 Field Sample Custody

All teams will ensure that samples, while in their possession, are maintained in a secure manner to prevent tampering, damage, or loss. At the conclusion of the sampling program, the team will relinquish all samples and FSDSs to the sample coordinator or designated secure sample storage area. The field team will be responsible for documenting this transfer of sample custody in the logbook.

B3.3 Chain-of-Custody Requirements

The chain-of-custody (COC) record is used as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. A complete COC record is required to accompany each shipment of samples. COC procedures will follow the requirements as stated in Site-specific SOP EPA-LIBBY-2012-06, *Sample Custody* (see **Appendix B**). The field sample coordinator will follow COC procedures to ensure proper sample custody between acceptance of the sample from the field teams to delivery or shipment to the laboratory.

A member of the sample coordination staff will manually enter sample information from the hard copy FSDS into the local Scribe field project database using a series of standardized data entry forms developed in Microsoft Access by ESAT, referred to as the sample Data Entry Tool, or the "DE Tool". The DE Tool has a variety of built-in QC functions that improve accuracy of data entry and help maintain data integrity. After the data entry is checked against the hard copy FSDSs (by a different sample coordination staff member than completed the original data entry), the DE Tool is used to prepare an electronic COC. A three-page carbon copy COC will be generated from the electronic COC. The field sample coordinator will retain one hard copy of the COC for the project file; the other two hard copies of the COC will accompany the sample shipment.

The field sample coordinator will note the analytical priority level for the samples (based on consultation with the LC) at the top of the COC. A copy of the investigation-specific Analytical Requirements Summary Sheet (see **Appendix D**) will also accompany each COC.

If any errors are found on a COC after shipment, the hard copy of the COC retained by the field sample coordinator will be corrected with a single strikeout, initial, and date. A copy of the corrected COC will be provided to the LC for distribution to the appropriate laboratory. It is the responsibility of the field data manager to make any corrections to the local Scribe field project database. Sample and COC information will be published to Scribe.NET regularly from the local Scribe field project database by the field data manager (see Section B10.1 for additional details).

B3.4 Sample Packaging and Shipping

Tissue samples will be hand-delivered to the CDM Smith office in Libby for subsequent shipment via overnight delivery to EMSL Analytical laboratory in Cinnaminson, New Jersey. Samples will be packaged and shipped in general accordance with SOP EPA-LIBBY-2012-07, *Packaging and Shipping of Environmental Samples* (see **Appendix B**). Tissue samples will be shipped frozen (fish) or on wet ice (game). Prior to sealing the shipping container, the field sample coordinator will sign the COC record and retain the bottom copy of the COC record for the project record.

B3.5 Holding Times

A holding time is defined as the allowable time between sample collection and analysis and/or extraction recommended to ensure accuracy and representativeness of analysis results, based on the nature of the analyte of interest and chemical stability factors. The holding time is calculated from the date and time of sample collection to the time of sample preparation and/or analysis. Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. In general, there are no holding time requirements for asbestos. Ashing the tissue samples will address any concerns with holding times for the medium.

B3.6 Archival and Final Disposition

All samples and grids will be maintained in storage at the analytical laboratory unless otherwise directed by the EPA. When authorized by the EPA, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

B4. Analytical Methods

B4.1 Analytical Methods and Requirements

This section discusses the analytical methods and requirements for tissue sampling program. Detailed information is included on the analysis of LA in the tissue samples, as well as sample preparation, and data reporting requirements.

An analytical requirements summary sheet (**TISSOU4-0812**), which details the specific preparation and analytical requirements associated with this sampling program, is provided in **Appendix D**. The analytical requirements summary sheet will be reviewed and approved by all participating laboratories in this sampling program prior to any sample handling. A copy of this analytical requirements summary sheet will be submitted with each COC record.

B4.1.1 Tissue Samples

Sample Preparation

Tissue samples will be prepared and analyzed in basic accordance with the procedures specified in SOP EPA-LIBBY-2012-13, *Analysis of Tissue for Asbestos* (see **Appendix B**). In brief, each sample is dried and ashed, and an aliquot of the resulting ash residue is acidified, suspended in water, and filtered. A total of three replicate filters will be created for each tissue sample using additional aliquots of the ash residue. Each filter will be used to prepare a minimum of three grids using the grid preparation techniques described in Section 9.3 of International Organization for Standardization (ISO) 10312:1995(E) (ISO 1995).

Analysis Method and Counting Rules

Grids will be examined by TEM using high magnification (~20,000x) in basic accordance with the recording procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications[6] LB-000016, LB-000029, LB-000066D, LB-000067, and LB-000085. In brief, all fibrous amphibole structures that have appropriate selective area electron diffraction (SAED) patterns and energy dispersive x-ray analysis (EDXA) spectra, and having a length greater than or equal to (≥) 0.5 µm and an aspect ratio (length: width) ≥ 3:1, will be recorded. If observed, chrysotile structures should be recorded using the same procedures. All LA structures longer than 0.5 µm will be recorded, even though the risk evaluation will utilize only structures longer than 10 µm.

[6] Copies of all Libby laboratory modifications are located in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab)

Stopping Rules

The stopping rules for the TEM of tissue samples are as follows:

1. Count a minimum of two grid openings from each of two grids.

2. Continue counting until one of the following is achieved:

    a. 25 LA structures have been observed.

    b. A total filter area of 0.25 square millimeters (mm^2) has been examined (this is approximately 25 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

The results for each tissue analysis will be expressed in terms of LA structures per gram (s/g) of tissue (dry weight). However, the laboratory should also report the sample-specific tissue mass pre- and post-drying to allow for the conversion of the concentrations to wet weight.

B4.1.2 Equipment Rinsate Water Samples

Sample Preparation

All equipment rinsate water samples (see Section B5.1.5) should be prepared for asbestos analysis in basic accordance with the techniques in EPA Method 100.2, as modified by Libby Laboratory Modification LB-000020A. In brief, all water samples will be prepared using an ozone/ultraviolet treatment that oxidizes organic matter that is present in the water or on the walls of the bottle, destroying the material that causes clumping and binding of asbestos structures. Following treatment, an aliquot of water (generally about 50 milliliters) will be filtered through a 25-millimeter diameter polycarbonate filter with a pore size of 0.1 µm with a mixed cellulose ester filter (0.45 µm pore size) used as a support filter.

Analysis Method and Counting Rules

Approximately one quarter of the filter will be used to prepare a minimum of three grids using the grid preparation techniques described in Section 9.3 of ISO 10312:1995(E). Grids will be examined by TEM in basic accordance with the recording procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications LB-000016, LB-000029, LB-000066D, LB-000067, and LB-000085.

All structures with fibrous morphology, an x-ray diffraction pattern consistent with amphibole asbestos, a energy dispersive spectrum consistent with LA, length ≥ 0.5 µm, and an aspect ratio (length:width) ≥ 3:1 will be counted and recorded. These counting rules will enable the

calculation of water concentrations based on both total LA and LA structures longer than 10 μm. If observed, chrysotile structures will be recorded, but chrysotile structure counting may stop after 25 structures have been recorded.

Stopping Rules

The TEM stopping rules for equipment rinsate water samples from this investigation are specified below and were selected to be consistent with the analytical requirements specified in other water sampling efforts conducted at the Site. The stopping rules are as follows:

1. Count a minimum of two grid openings from each of two grids.
2. Continue counting until one of the following is achieved:
    a. The target analytical sensitivity of 50,000 L^{-1} has been achieved.
    b. 25 LA structures have been observed.
    c. A total filter area of 1.0 mm^2 has been examined (this is approximately 100 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

B4.2 Analytical Data Reports

An analytical data report will be prepared by the laboratory and submitted to the LC after the completion of all required analyses within a specific laboratory job (or sample delivery group). This analytical data report may vary by laboratory and analytical method but generally includes a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include copies of the signed COC forms, analytical data summaries, a QC package, and raw data. Raw data is to consist of instrument preparation logs, instrument printouts, and QC sample results including, instrument maintenance records, COC check in and tracking, raw data instrument print outs of sample results, analysis run logs, and sample preparation logs. The laboratory will provide an electronic scanned copy of the analytical data report to the LC and others, as directed by the LC.

B4.3 Laboratory Data Reporting Tools

Standardized data reporting tools (i.e., EDDs) have been developed specifically for the Libby project to ensure consistency between different laboratories in the presentation and submittal of analytical data. In general, unique Libby-specific EDDs have been developed for each analytical method and each medium. Since the beginning of the Libby project, each EDD has undergone continued development and refinement to better accommodate current and anticipated future data needs and requirements. EDD refinement continues based on laboratory and data user input. Electronic copies of all current EDD templates are provided in the Libby Lab eRoom.

For TEM analyses, detailed raw structure data will be recorded and results will be transmitted using the Libby-specific EDDs[7] for TEM reporting of tissue data. Standard project data reporting requirements will be met for TEM analyses. EDDs will be transmitted electronically (*via* email) to the following:

- Doug Kent, Kent.Doug@epa.gov
- Janelle Lohman, Lohman.Janelle@epa.gov
- Tracy Dodge, DodgeTA@cdmsmith.com
- Phyllis Haugen, HaugenPJ@cdmsmith.com
- Libby project email address for CDM Smith, libby@cdmsmith.com

Note: ESAT is in the process of developing a new Site-specific analytical results reporting tool, referred to as the Libby Asbestos Data Tool (LADT). This tool is a relational Microsoft® Access database with a series of standard data entry forms specific to each analytical method. The LADT creates a Microsoft® Excel export file that can be directly uploaded into an analytical Scribe project database (see Section B10.3). Laboratories have the option of using LADT as a data reporting method instead of the Libby-specific EDDs.

B4.4 Analytical Turn-around Time

Analytical turn-around time will be negotiated between the LC and the laboratory. It is anticipated that turn-around times of 2-4 weeks are acceptable, but this may be revised as determined necessary by the EPA.

B4.5 Custody Procedures

Specific laboratory custody procedures are provided in each laboratory's *Quality Assurance Management Plan*, which have been independently reviewed at the time of laboratory procurement. While specific laboratory sample custody procedures may differ between laboratories, the basic laboratory sample custody process is described briefly below.

Upon receipt at the facility, each sample shipment will be inspected to assess the condition of the shipment and the individual samples. This inspection will include verifying sample integrity. The accompanying COC will be cross-referenced with all of the samples in the shipment. The laboratory sample coordinator will sign the COC and maintain a copy for their project files.

Depending upon the laboratory-specific tracking procedures, the laboratory sample coordinator may assign a unique laboratory identification number to each sample on the COC. This number, if assigned, will identify the sample through all further handling at the laboratory. It is the

[7] The most recent version of the TEM EDDs for the Libby project are provided in the Libby Lab eRoom.

responsibility of the laboratory manager to ensure that internal logbooks and records are maintained throughout sample preparation, analysis, and data reporting.

B5. Quality Assurance/Quality Control

B5.1 Field

Field QA/QC activities include all processes and procedures that have been designed to ensure that field samples are collected and documented properly, and that any issues/deficiencies associated with field data collection or sample processing are quickly identified and rectified. The following sections describe each of the components of the field QA/QC program implemented at the Site.

B5.1.1 Training

Before performing field work in Libby, field personnel are required to read all governing field guidance documents relevant to the work being performed and attend a field planning meeting specific to this sampling effort. Additional information on field training requirements is provided in Section A8.1.

B5.1.2 Modification Documentation

All field deviations from and modifications to this SAP/QAPP will be recorded on the Libby field ROM form[8]. The field ROM forms will be used to document all permanent and temporary changes to procedures contained in guidance documents governing investigation work that have the potential to impact data quality or usability. Any minor deviations (i.e., those that will not impact data quality or usability) will be documented in the field logbooks. ROMs are completed by the FTL overseeing the investigation/activity, or by assigned field or technical staff. As modifications to governing documents are implemented, the FTL will communicate the changes to the field teams conducting activities associated with the modification.

Each completed field ROM is assigned a unique sequential number (e.g., LFO-000026) by the CDM Smith field QAM. A ROM tracking log for all field modifications is maintained by the field QAM. This tracking log briefly describes the ROM being documented, as well as ROM author, the reviewers, and date of approval. Once a form is prepared, it is submitted to the appropriate EPA RPM for review and approval. Copies of approved ROMs are available in the CDM Smith field office server in Libby.

[8] The most recent version of the field ROM form is provided in the Libby Field eRoom.

B5.1.3 Field Surveillances

Field surveillances consist of periodic observations made to evaluate continued adherence to investigation-specific governing documents. It is not anticipated that field surveillance will be performed for this investigation. However, field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies.

B5.1.4 Field Audits

Field audits are broader in scope than field surveillances. Audits are evaluations conducted by qualified technical or QA staff that are independent of the activities audited. Field audits can be conducted by field contractors, internal EPA staff, or EPA contracted auditors. It is the responsibility of the EPA RPM to ensure that field auditing requirements are met for each investigation. Because of the opportunistic nature of this sampling event, it is not anticipated that any field audits would be performed as part of this investigation.

B5.1.5 Field QC Samples

Field QC samples are collected to help ensure that field samples are not contaminated from exogenous sources during sample collection, and to help evaluate the precision of field sample analytical results. Field QC samples are assigned unique field identifiers and are submitted to the analytical laboratory along with the associated field samples. Three types of field QC samples are possible for the tissue sampling as described in the following sections:

Field Blanks

Field blanks are collected to evaluate potential contamination introduced during sample collection, shipping and handling, or analysis. For this sampling effort, a total of three field blanks will be submitted, one for each type of animal (deer, elk, fish). Field blanks for game tissue will be collected as steak from a local supermarket. Field blanks for fish tissue will be collected as farm-raised trout from a local supermarket. The field blanks for game will be handled in the same manner as the tissue samples collected and will be subjected to the collection process at the same time as the gross necropsy. The field blanks will be submitted to the analytical laboratory and analyzed for asbestos by TEM analysis using the same procedures as the field samples (see Section B4.1.1).

If any asbestos is observed in a field blank, the FTL and/or laboratory manager will be notified and will take appropriate measures to ensure staff are employing proper sample handling techniques. In addition, a qualifier of "FB" will be added to the related field sample results in the project database to denote that the associated field blank had asbestos structures detected.

Field Duplicates

One field duplicate sample will be collected of muscle from each animal (deer and elk). The tissue field duplicate should be collected as one half of the flank muscle tissue collected. One field duplicate will be collected for fish, representing the remaining fillet for one selected fish.

It is the responsibility of the FTL to ensure that the field duplicates are collected at the specified frequency. The field duplicate is given a unique sample number, and field personnel will record the sample number of the associated co-located sample in the parent sample number field of the FSDS. The same station location is assigned to the field duplicate sample as the parent field sample. Field duplicates will be sent for analysis by the same method as field samples (see Section B4.1.1) and are blind to the laboratory.

Field duplicate results will be compared to the original parent field sample using the Poisson ratio test using a 90% confidence interval (CI) (Nelson 1982). Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, typically, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability.

Equipment Rinsates

Equipment rinsates are collected to evaluate potential contamination that arises to due inadequate decontamination of sampling equipment. Equipment rinsates will only be collected if dedicated field sampling equipment (i.e., scalpels, scissors and other dissection instruments) is not used. If dedicated equipment is not used, following decontamination efforts, the decontaminated equipment should be rinsed with clean water (e.g., store-bought drinking water), and the resulting rinsate collected in a 500-milliliter high-density polyethylene container (leaving headspace at the top of the container). At least one equipment rinsate blank should be collected per equipment decontamination effort. It is the responsibility of each field team to collect the appropriate number of equipment rinsate blanks. Equipment rinsate blanks should be labeled with a unique sample number and submitted for analysis by TEM (see Section B4.1.2).

If any asbestos structures are observed in an equipment rinsate, the FTL and/or laboratory manager will be notified and will take appropriate measures to ensure staff are employing proper sample handling techniques. In addition, a qualifier of "EB" will be added to the related field sample results in the project database to denote that the associated equipment rinsate had asbestos structures detected.

B5.2 Analytical Laboratory

Laboratory QA/QC activities include all processes and procedures that have been designed to ensure that data generated by an analytical laboratory are of high quality and that any problems

in sample preparation or analysis that may occur are quickly identified and rectified. The following sections describe each of the components of the analytical laboratory QA/QC program implemented at the Site.

B5.2.1 Training/Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Additional information on laboratory training and certification requirements is provided in Section A8.2.

Laboratories handling samples collected as part of this sampling program will be provided a copy of and will adhere to the requirements of this SAP/QAPP. Samples collected under this SAP/QAPP will be analyzed in accordance with standard EPA and/or nationally-recognized analytical procedures (i.e., Good Laboratory Practices) in order to provide analytical data of known quality and consistency.

B5.2.2 Modification Documentation

All deviations from project-specific and method guidance documents will be recorded on the laboratory ROM form[9]. The ROM will be used to document all permanent and temporary changes to analytical procedures. ROMs will be completed by the appropriate laboratory or technical staff. As ROMs are completed, it is the responsibility of the LC to communicate any changes to the project laboratories. When the project management team determines the need, this SAP/QAPP will be revised to incorporate necessary modifications. Copies of approved ROMs for this SAP/QAPP will be made available in the Libby Lab eRoom.

B5.2.3 Laboratory Audits

Each laboratory working on the Libby project is required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. These audits are performed by EPA personnel (and their contractors), that are external to and independent of, the Libby laboratory team members. These audits ensure that each analytical laboratory meets the basic capability and quality standards associated with analytical methods for asbestos used at the Libby site. They also provide information on the availability of sufficient laboratory capacity to meet potential testing needs associated with the Site.

<u>External Audits</u>

Audits consist of several days of technical and evidentiary review of each laboratory. The technical portion of the audit involves an evaluation of laboratory practices and procedures associated with the preparation and analysis of samples for the identification of asbestos. The

[9] The most recent version of the laboratory ROM form is available in the Libby Lab eRoom.

evidentiary portion of the audit involves an evaluation of data packages, record keeping, SOPs, and the laboratory *QA Management Plan*. A checklist of method-specific requirements for the commonly used methods for asbestos analysis is prepared by the auditor prior to the audit, and used during the on-site laboratory evaluation.

Evaluation of the capability for a laboratory to analyze a sample by a specific method is made by observing analysts performing actual sample analyses and interviewing each analyst responsible for the analyses. Observations and responses to questions concerning items on each method-specific checklist are noted. The determination as to whether the laboratory has the capability to analyze a sample by a specific method depends on how well the analysts follow the protocols detailed in the formal method, how well the analysts follow the laboratory-specific method SOPs, and how the analysts respond to method-specific questions.

Evaluation of the laboratory to be sufficient in the evidentiary aspect of the audit is made by reviewing laboratory documentation and interviewing laboratory personnel responsible for maintaining laboratory documentation. This includes personnel responsible for sample check-in, data review, QA procedures, document control, and record archiving. Certain analysts responsible for method quality control, instrument calibration, and document control are also interviewed in this aspect of the audit. Determination as to the capability to be sufficient in this aspect is made based on staff responses to questions and a review of archived data packages and QC documents.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report for each analytical laboratory participating in the Libby program. These reports are handled as business confidential items. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each laboratory to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

It is the responsibility of the QATS contractor to prepare an On-Site Audit Trend Analysis Report on an annual basis. This report shall include a compilation and trend analysis of the on-site audit findings and recommendations. The purpose of this reported is to identify common asbestos laboratory performance problems and isolate the potential causes.

Internal Audits

Each laboratory will also conduct periodic internal audits of their specific operations. Details on these internal audits are provided in the laboratory *QA Management Plan*. The laboratory QAM should immediately contact the LC and the QATS contractor if any issues are identified during internal audits that may impact data quality for OU3 samples.

B5.2.4 Laboratory QC Analyses

General TEM QC Requirements

The Libby-specific QC requirements for TEM analyses of asbestos are patterned after the requirements set forth by NVLAP. In brief, there are three types of laboratory-based QC analyses for TEM – laboratory blanks, recounts, and repreparations. Detailed information on the Libby-specific requirements for each type of TEM QC analysis, including the minimum frequency rates, selection procedures, acceptance criteria, and corrective actions are provided in the most recent version of Libby Laboratory Modification LB-000029.

With the exception of inter-laboratory analyses, it is the responsibility of the laboratory manager to ensure that the proper number of TEM QC analyses are completed. Inter-laboratory analyses for TEM will be selected *post hoc* by the QATS contractor or their designate in accordance with the selection procedures presented in LB-000029. The LC will provide the list of selected inter-laboratory analyses to the laboratory manager and will facilitate the exchange of samples between the analytical laboratories.

Tissue-specific QC Requirements

In addition to the laboratory-based QC analyses discussed above, TEM analyses of tissues have additional QC analyses that are required, including drying blanks and filtration blanks. Because three replicate filters will be prepared and analyzed for each tissue sample, no laboratory duplicate analyses will be required for this sampling effort. Detailed information on the Libby-specific requirements for each type of TEM QC analysis is provided in the SOP EPA-LIBBY-2012-13 (see **Appendix B**). It is the responsibility of the laboratory manager to ensure that the proper number of TEM QC analyses is completed.

B6/B7. Instrument Maintenance and Calibration

B6/B7.1 Field Equipment

All field equipment (e.g., GPS units) should be maintained in basic accordance with manufacturer specifications. When a piece of equipment is found to be operating incorrectly, the piece of equipment will be labeled "out of order" and placed in a separate area from the rest of the sampling equipment. The person who identified the equipment as "out of order" will notify the FTL overseeing the investigation activities. It is the responsibility of the FLT to facilitate repair of the out-of-order equipment. This may include having appropriately trained field team members complete the repair or shipping the malfunctioning equipment to the manufacturer. Field team members will have access to basic tools required to make field acceptable repairs. This will ensure timely repair of any "out of order" equipment.

B6/B7.2 Laboratory Instruments

All laboratory instruments used for this project will be maintained and calibrated in accordance with the manufacturer's instructions. If any deficiencies in instrument function are identified, all analyses shall be halted until the deficiency is corrected. The laboratory shall maintain a log that documents all routine maintenance and calibration activities, as well as any significant repair events, including documentation that the deficiency has been corrected.

B8. Inspection/Acceptance of Supplies and Consumables

B8.1 Field

In advance of field activities, the FTL will check the field equipment/supply inventory and procure any additional equipment and supplies that are needed. The FTL will also ensure any in-house measurement and test equipment used to collect data/samples as part of this SAP/QAPP is in good, working order, and any procured equipment is acceptance tested prior to use. Any items that the FTL determines unacceptable will be removed from inventory and repaired or replaced as necessary.

The following list summarizes the general equipment and supplies required for most investigations:

- Field logbook – Used to document field sampling activities and any problems in sample collection or deviations from this SAP/QAPP. See Section B3.1.3 for standard procedures for field logbooks.

- FSDSs – FSDSs are medium-specific forms that are used to document sample details (i.e., sampling location, sample number, medium, field QC type, etc.). See Section B3.1.2 for standard procedures for the completion of FSDSs.

- Sample ID number labels – Sample numbers are sequential numbers with investigation-specific prefixes. Sample number labels are pre-printed and checked out to the field teams by the FTL or their designate. To avoid potential transcription errors in the field, multiple labels of the same sample number are prepared – one label is affixed to the collected sample, one label is affixed to the hard copy FSDS form. Labels may also be affixed to the field logbook. See Section B3.1.1 for additional information on sample labels.

- Indelible ink pen, permanent marker – Indelible ink pens are used to complete required manual data entry of information on the FSDS and in the field logbook (pencil may not be used). Permanent markers may also be used to write sample numbers on the sample containers.

- Personal Protective Equipment (PPE) - As required by the HASP.

- Land survey map or aerial photo – Used to identify appropriate sampling locations. In some cases, sketches may be added to the map/photo to designate sampling and visual inspection locations and other site features.

- Digital camera – Used to document sampling locations and conditions. See Section B3.1.4 for standard procedures in photographic documentation.

- GPS unit– Used to identify and mark sampling locations. See Section B2.2 for standard procedures in GPS documentation.

- Zip-top bags – Zip-top bags are used as sample containers for most types of environmental samples. Sample number labels will be affixed to the bags or the sample number will be hand-written in permanent marker on the bags.

- Decontamination equipment – Used to remove any residual asbestos contamination on reusable sampling equipment between the collection of samples. See Section B2.3 for standard decontamination procedures.

In addition to the generic equipment list, the following equipment will be required for sampling activities as part of this program:

- Baiting Supplies - including corn, grains and some commercially available products
- Game/trail camera – for animal surveillance
- Rifle - 300 Winchester Magnum (owned by Michael Cirian)
- ATV
- Tarps and rope
- Tissue dissection materials – cutting boards, dissecting trays, fillet knives, forceps, bone saw, pliers, large scissors, scalpel, and knife sharpener

B8.2 Laboratory

The laboratory manager is responsible for ensuring that all reagents and disposable equipment used in this project is free of asbestos contamination. This is demonstrated by the collection of laboratory blank samples, as described in Section B5.2.4.

B9. Non-direct Measurements

There are no non-direct measurements that will be used for this sampling program.

B10. Data Management

The following subsections describe the field and analytical laboratory data management procedures and requirements for this investigation. These subsections also describe the project databases utilized to manage and report data from this investigation. Detailed information regarding data management procedures and requirements can be found in the *EPA Data Management Plan* for the Libby Asbestos Superfund Site (EPA 2012b).

B10.1 Field Data Management

Scribe is a software tool developed by ERT to assist in the process of managing environmental data. A Scribe project is a Microsoft Access database. Data for the Site are captured in various Scribe projects. Additional information regarding Scribe and the Libby Scribe project databases is discussed in Section B10.3.

The field data manager utilizes a "local" field Scribe project database (i.e., LibbyCDM_Field.mdb) to maintain field sample information. The term "local" denotes that the database resides on the server or personal computer of the entity that is responsible for the creating/managing the database. It is the responsibility of the field data manager to ensure that all local field Scribe project databases are backed-up nightly to a local server.

Field sample information from the FSDS is manually entered by a member of the field sample coordination staff using a series of standardized data entry forms (i.e., DE Tool). This tool is a Microsoft Access database that was originally developed by ESAT. The DE Tool is currently maintained by CDM Smith and resides on the local server in the Libby field office. This tool is used to prepare an electronic COC. Data in the DE Tool are imported into the local field Scribe project database by the field data manager.

It is the responsibility of the field data manager to "publish" sample and COC information from the local field Scribe database to Scribe.NET on a daily basis. It is not until a database has been published via Scribe.NET that it becomes available to external users.

B10.2 Analytical Laboratory Data Management

The analytical laboratories utilize several standardized data reporting tools developed specifically for the Libby project to ensure consistency between laboratories in the presentation and submittal of analytical data. In general, a unique Libby-specific EDD has been developed for each analytical method and each sampling medium. Electronic copies of all current EDD templates are provided in the Libby Lab eRoom.

Once the analytical laboratory has populated the EDD with results, the spreadsheet(s) are transmitted via email to the ESAT TEM Laboratory Manager, the ESAT project data manager,

and the FTL (or their designate). (Other email recipients may also be specified by the ESAT LC).

The ESAT project database manager utilizes a local analytical Scribe project database (i.e., LibbyLab2012.mdb) to maintain analytical results information. The EDDs are uploaded directly into the analytical Scribe project database. It is the responsibility of the ESAT project data manager to publish analytical results information from the local analytical Scribe database to Scribe.NET.

B10.3 Libby Project Database

As noted above, Scribe is a software tool developed by ERT to assist in the process of managing environmental data. A Scribe project is a Microsoft Access database. Multiple Scribe projects can be stored and shared through Scribe.NET, which is a web-based portal that allows multiple data users controlled access to Scribe projects. Local Scribe projects are "published" to Scribe.NET by the entity responsible for managing the local Scribe project. External data users may "subscribe" to the published Scribe projects via Scribe.NET to access data. Subscription requests are managed by ERT.

All data collected for this investigation will be maintained in Scribe. As discussed above, data will be are captured in various Scribe project databases, including a field Scribe project (i.e., LibbyCDM_Field.mdb) and an analytical results Scribe project (i.e., LibbyLab2012.mdb).

B10.4 Data Reporting

Data users can access data for the Libby project through Scribe.NET. To access data, a data user must first download the Scribe application from the EPA ERT website[10]. The data user must then subscribe to each of the published Scribe projects for the Site using login and password information that are specific to each individual Scribe project. Scribe subscriptions for the Libby project are managed by ERT. Using the Scribe application, a data user may download a copy of any published Scribe project database to their local hard drive. It is the responsibility of the data user to regularly update their local copies of the Libby Scribe projects via Scribe.NET.

The Scribe application provides several standard queries that can be used to summarize and view results within an individual Scribe project. However, these standard Scribe queries cannot be used to summarize results across multiple Scribe projects (e.g., it is not possible to query both the "LibbyCDM_Field" project and the "LibbyLab2012" project using these standard Scribe queries).

If data users wish to summarize results across multiple published Scribe projects, there are two potential options. Data users may request the development of a "combined" project from ERT. This combined project compiles tables from multiple published Scribe projects into a single

[10] http://www.ertsupport.org/scribe_home.htm

Scribe project. This allows data users to utilize the standard Scribe queries to summarize and view results.

Alternatively, data users may download copies of multiple published Scribe project databases for the Site and utilize Microsoft Access to create user-defined queries to extract the desired data across Scribe projects. This requires that the data user is proficient in Microsoft Access and has an intimate knowledge of proper querying methods for asbestos data for the Site.

It is the responsibility of the data users to perform a review of results generated by any data queries and standard reports to ensure that they are accurate, complete, and representative. If issues are identified by the data user, they should be reported to the EPA Region 8 Data Manager via email (Mosal.Jeffrey@epa.gov). It is the responsibility of the EPA Region 8 Data Manager to notify the appropriate entity (e.g., field, analytical laboratory) in order to rectify the issue. A follow-up email will be sent to the party reporting the issue to serve as confirmation that a resolution has been reached and any necessary changes have been made.

C Assessment and Oversight

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities.

C1. Assessment and Response Actions

C1.1 Assessments

System assessments are qualitative reviews of different aspects of project work to check the use of appropriate QC measures and the general function of the QA system. Field and office system assessments will be performed under the direction of CDM Smith's QA Director, with support from the CDM Smith QAM. As noted previously, it is not anticipated that any field surveillances or audits will be performed during this sampling program. However, field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies.

Laboratory system assessments/audits will be coordinated by the EPA. Performance assessments for the laboratories may be accomplished by submitting blind reference material (i.e., performance evaluation samples). These assessment samples are samples with known concentrations that are submitted to the laboratories without identifying them as such to the laboratories. Performance assessments will be coordinated by the EPA.

C1.2 Response Actions

Corrective response actions will be implemented on a case-by-case basis to address quality problems. Minor actions taken to immediately correct a quality problem will be documented in the applicable field or laboratory logbooks and a verbal report will be provided to the appropriate manager (e.g., the FTL or EPA LC). Major corrective actions will be approved by the EPA Remedial Project Manager and the appropriate manager prior to implementation of the change. Major response actions are those that may affect the quality or objective of the investigation. EPA project management will be notified when quality problems arise that cannot be corrected quickly through routine procedures.

In addition, when modifications to this SAP/QAPP are required, either for field or laboratory activities, a ROM must be completed by field staff and approved by the EPA prior to implementation.

C2. Reports to Management

No regularly-scheduled written reports to management are planned as part of this project. However, QA reports will be provided to management for routine audits and whenever quality problems are encountered. Field staff will note any quality problems on FSDSs or in field logbooks. Further, the CDM Smith project manager will inform EPA project management upon encountering quality issues that cannot be immediately corrected. Weekly reports and change request forms are not required for work performed under this SAP/QAPP.

D Data Validation and Usability

D1. Data Review, Verification and Validation

D1.1 Data Review

Data review of Scribe project data typically occurs at the time of data reporting by the data users and includes cross-checking that sample IDs and sample dates have been reported correctly and that calculated analytical sensitivities or reported values are as expected. If discrepancies are found, the data user will contact the EPA database administrator, who will then notify the appropriate entity (field, preparation facility, or laboratory) in order to correct the issue.

D1.2 Criteria for LA Measurement Acceptability

Several factors are considered in determining the acceptability of LA measurements in samples analyzed by TEM. This includes the following:

1. *Evenness of filter loading.* This is evaluated using a chi-squared (CHISQ) test, as described in Annex F2 of ISO 10312. If a filter fails the CHISQ test for evenness, the result may not be representative of the true concentration in the sample, and the result should be given low confidence.

2. *Results of QC samples.* This includes both field and laboratory QC samples, such as field and laboratory blank samples, as well as various types of recount and re-preparation analyses. If significant LA contamination is detected in field or laboratory blanks, all samples prepared on that day should be considered to be potentially biased high. If agreement between original analyses and field or laboratory duplicates (i.e., repreparation or recount analyses) is poor, results for those samples should be given low confidence.

D2. Verification and Validation Methods

D2.1 Data Verification

Data verification includes checking that results have been transferred correctly from the original hand-written, hard copy field and analytical laboratory documentation to the project databases. The goal of data verification is to identify and correct data reporting errors.

For analytical laboratories that utilize the Libby-specific EDD spreadsheets, data checking of reported analytical results begins with automatic QC checks that have been built into the spreadsheets. In addition to these automated checks, a detailed manual data verification effort

will be performed for 10% of all samples and TEM analytical results collected as part of this sampling effort. This data verification process utilizes Site-specific SOPs (see **Appendix B**) developed to ensure TEM results and field sample information in the project databases is accurate and reliable:

- EPA-LIBBY-09 – *SOP for TEM Data Review and Data Entry Verification* – This Site-specific SOP describes the steps for the verification of TEM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-11 – *SOP for FSDS Data Review and Data Entry Verification* – This Site-specific SOP describes the steps for the verification of field sample information, based on a review of the FSDS form, and verification of the transfer of results from the FSDS forms into the project database. An FSDS review is performed on all samples selected for TEM data verification.

The data verification review ensure that any data reporting issues are identified and rectified to limit any impact on overall data quality. If issues are identified during the data verification, the frequency of these checks may be increased as appropriate.

Data verification will be performed by appropriate technical staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements. The data verifier will prepare a data verification report (template reports are included in the SOPs) to summarize any issues identified and necessary corrections. A copy of this report will be provided to the appropriate project data manager, LC, and the EPA RPM. The data verifier will also transmit the results of the data verification, including any electronic files summarizing identified discrepancies, to the EPA Region 8 Data Manager via email for resolution. A follow-up email will be sent to the party reporting the issue to serve as confirmation that a resolution has been reached.

It is the responsibility of the EPA Region 8 Data Manager to coordinate with the FTL and/or LC to resolve any project database corrections and address any recommended field or laboratory procedural changes from the data verifier. The EPA Region 8 Data Manager is also responsible for electronically tracking in the project database which data have been verified, who performed the verification, and when.

D2.2 Data Validation

Unlike data verification, where the goal is to identify and correct data reporting errors, the goal of data validation is to evaluate overall data quality and to assign data qualifiers, as appropriate, to alert data users to any potential data quality issues. Data validation will be performed by the QATS contractor (or their designate), with support from technical support

staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements.

Data validation for asbestos should be performed in basic accordance with the draft *National Functional Guidelines (NFG) for Asbestos Data Review* (EPA 2011), and should include an assessment of the following:

- Internal and external field audit/surveillance reports
- Field ROMs
- Field QC sample results
- Internal and external laboratory audit reports
- Laboratory contamination monitoring results
- Laboratory ROMs
- Internal laboratory QC analysis results
- Inter-laboratory analysis results
- Performance evaluation results
- Instrument checks and calibration results
- Data verification results (i.e., in the event that the verification effort identifies a larger data quality issue)

A comprehensive data validation effort should be completed quarterly and results should be reported as a technical memorandum. This technical memorandum shall detail the validation procedures performed and provide a narrative on the quality assessment for each type of asbestos analysis, including the data qualifiers assigned, and the reason(s) for these qualifiers. The technical memorandum shall detail any deficiencies and required corrective actions.

The QATS contractor will also prepare an annual addendum to the *Quality Assurance and Quality Control Summary Report for the Libby Asbestos Superfund Site* (CDM Smith 2011) to summarize results of the quarterly data validation efforts. This addendum should include a summary of any data qualifiers that are to be added to the project database to denote when results do not meet NFG guidelines and/or project-specific acceptance criteria. This addendum should also include recommendations for Site QA/QC program changes to address any data quality issues.

The data validator will complete and transmit the results of each data validation effort to the EPA Region 8 Data Manager via email. This email should include a summary of the records that have been validated, the date they were validated, any recommended data qualifiers, and their associated reason codes. It is the responsibility of the EPA Region 8 Data Manager to ensure that the appropriate data qualifiers and reason codes recommended by the data validator are added to the project database, and to electronically track in the project database which data have been validated, who performed the validation, and when.

In addition to performing quarterly data validation efforts, it is the responsibility of the QATS contractor (or their designate) to perform regular evaluations of all field blanks and laboratory blanks, to ensure that any potential contamination issues are quickly identified and resolved. If any blank contamination is noted, the QATS contractor should immediately contact the appropriate field QAM or SPF QAM to ensure that corrective actions are made.

D3. Reconciliation with User Requirements

It is the responsibility of data users to perform a data usability assessment to ensure that DQOs have been met, and reported investigation results are adequate and appropriate for their intended use. This data usability assessment should utilize results of the data verification and data validation efforts to provide information on overall data quality specific to each investigation.

The data usability assessment should evaluate results with regard to several data usability indicators. **Table D-1** summarizes several indicators of data usability and presents general evaluation methods for each indicator. Depending upon the nature of the investigation, other evaluation methods may also be appropriate. The data usability assessment results and conclusions should be included in any investigation-specific data summary reports.

Non-attainment of project requirements may result in additional sample collection or field observations in order to achieve project needs.

References

Amandus, H.E. and Wheeler R. 1987. The Morbidity and Mortality of Vermiculite Miners and Millers Exposed to Tremolite-Actinolite: Part II. Mortality. *Am. J. Ind. Med*. 11:15-26.

CDM Smith (formerly CDM). 2011. Quality Assurance and Quality Control Summary Report for the Libby Asbestos Superfund Site. Prepared for U.S. Environmental Protection Agency, Region 8 by CDM Smith. Draft – May.

EPA (U.S. Environmental Protection Agency). 2001. *EPA Requirements for Quality Assurance Project Plans – EPA QA/R-5*. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-01/003. March. http://www.epa.gov/quality/qs-docs/r5-final.pdf

_____. 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4*. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-06/001. February. http://www.epa.gov/quality/qs-docs/g4-final.pdf

_____. 2008a. Performance Evaluation of Laboratory Methods for the Analysis of Asbestos in Soil at the Libby, Montana Superfund Site. Produced by Syracuse Research Corporation for U.S. Environmental Protection Agency, Region 8. Draft – October 7.

_____. 2008b. Characteristic EDS Spectra for Libby-Type Amphiboles. Produced by Syracuse Research Corporation for U.S. Environmental Protection Agency, Region 8. Final – March 18.

_____. 2011. *National Functional Guidelines for Asbestos Data Review.* U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation. Draft – August.

_____. 2012a. *Phase V Sampling and Analysis Plan for Operable Unit 3, Libby Asbestos Superfund Site – Part B: 2012 Ecological Studies*. Produced by CDM Smith for U.S. Environmental Protection Agency, Region 8. Revision 2 – July.

_____. 2012b. EPA Data Management Plan, Libby Asbestos Superfund Site. U.S. Environmental Protection Agency, Emergency Response DATA Team. Draft – January 18.

Foresman, K.R. 2001. *The Wild Mammals of Montana*. Special Publication 12. The American Society of Mammalogists. 278 pp.

ISO (International Organization for Standardization). 1995. *Ambient Air – Determination of asbestos fibers – Direct-transfer transmission electron microscopy method.* ISO 10312:1995(E).

Jensen, W. 1999. Aging Elk. *North Dakota Outdoors* 62(2):16-20. Jamestown, ND: Northern Prairie Wildlife Research Center Online. http://www.npwrc.usgs.gov/resource/mammals/agelk/index.htm

McDonald, J.C.; McDonald, A.D.; Armstrong, B.; and Sebastien, P. 1986. Cohort study of mortality of vermiculite miners exposed to tremolite. *Brit. J. Ind. Med*. 43:436-444.

Nelson, W. 1982. *Applied Life Data Analysis*. John Wiley & Sons, New York. pp 438-446.

NTP (National Toxicology Program). 1985. Technical report series no. 295. *Toxicology and carcinogenesis studies of chrysotile asbestos (CAS No. 12001-29-5) in F344/N rats (feed studies)*. Research Triangle Park, NC: U.S. Department of Health and Human Services, Public Health Service, National Institutes of Health. NIH Publication No. 86-2551.

Peipins, L.A.; Lewin, M.; Campolucci, S.; Lybarger, J.A.; Miller, A.; Middleton, D. et al. 2003. Radiographic abnormalities and exposure to asbestos-contaminated vermiculite in the community of Libby, Montana, USA. *Environmental Health Perspectives*. 111:1753-1759.

Sullivan, P.A. 2007. Vermiculite, Respiratory Disease and Asbestos Exposure in Libby, Montana: Update of a Cohort Mortality Study. *Environmental Health Perspectives* doi:10.1289/ehp.9481 available online at http://dx.doi.org.

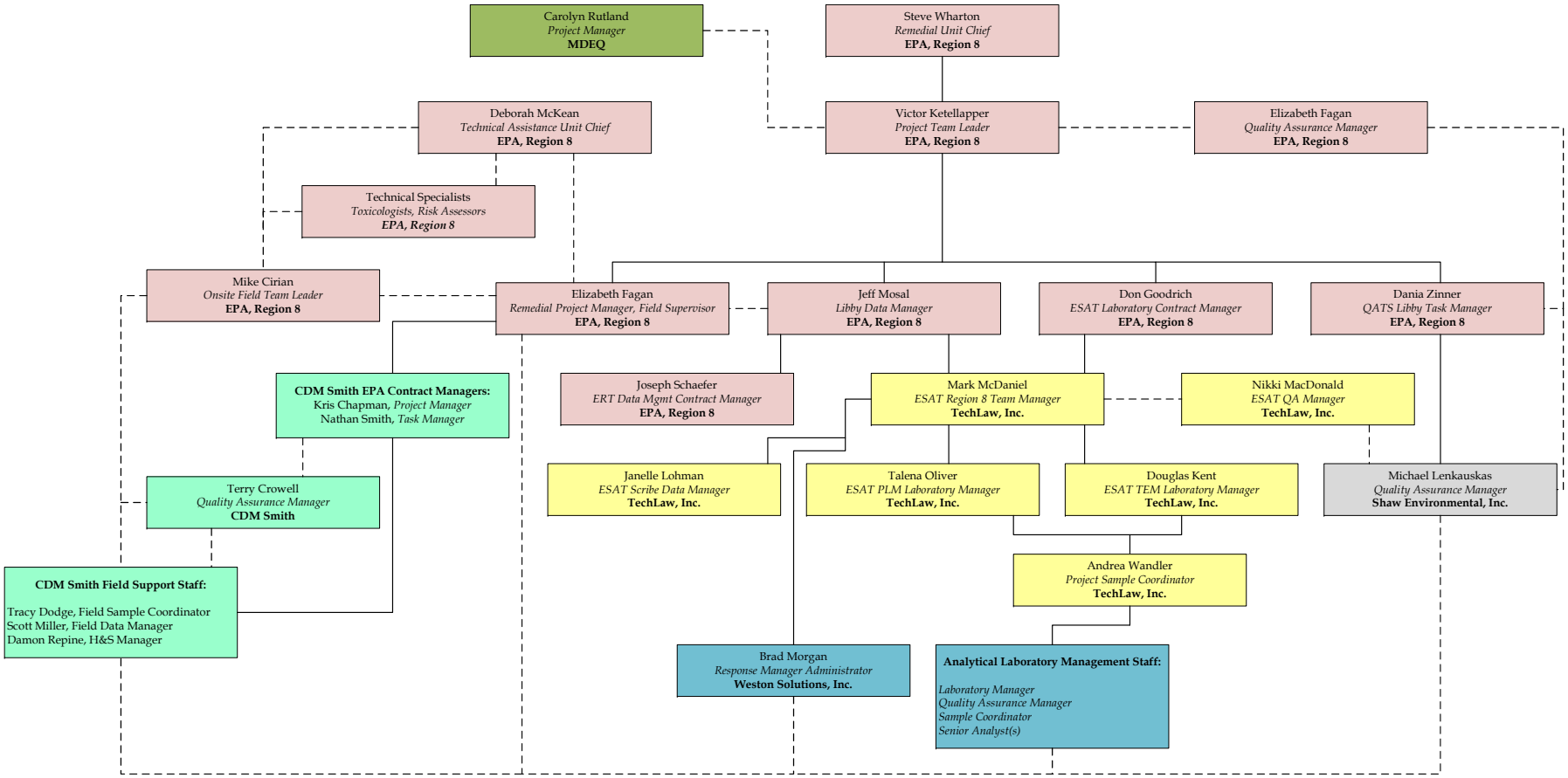

Figure A-1. Organizational Chart for the Fish & Game Tissue Sampling Program

EPA Region 8 Staff
CDM Smith Staff
Shaw Staff
Lines of authority
USACE Staff
TechLaw Staff
Lines of communication
MDEQ Staff
TechLaw Subcontractors

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Libby Asbestos Superfund Site
Operable Unit 3 (Study Area)

OU 3 (Study Area) - Mine & Kootenai River

Date: October 7, 2010

Map Projection: UTM, Meters, Zone 11N, NAD83

Data Sources: *Operable Units* - U.S. EPA Region 8 (2010);
Imagery - 1-meter USDA NAIP (2009).

0 1 2 Miles

0 1 2 Kilometers

N

EPA REGION 8 serving the states & tribes of Colorado, Montana, Wyoming, Utah, North Dakota & South Dakota

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

OU 3

2

Troy

Kootenai River

OU 3

2

Quartz Creek

Cedar Creek

Pipe Creek Rd

Kootenai River Rd

37

Kootenai River

OU 3

2

Purcell Creek

Flower Creek

Libby

Rainey Creek

OU 3

Lake Koocanusa

37

Kootenai River

2

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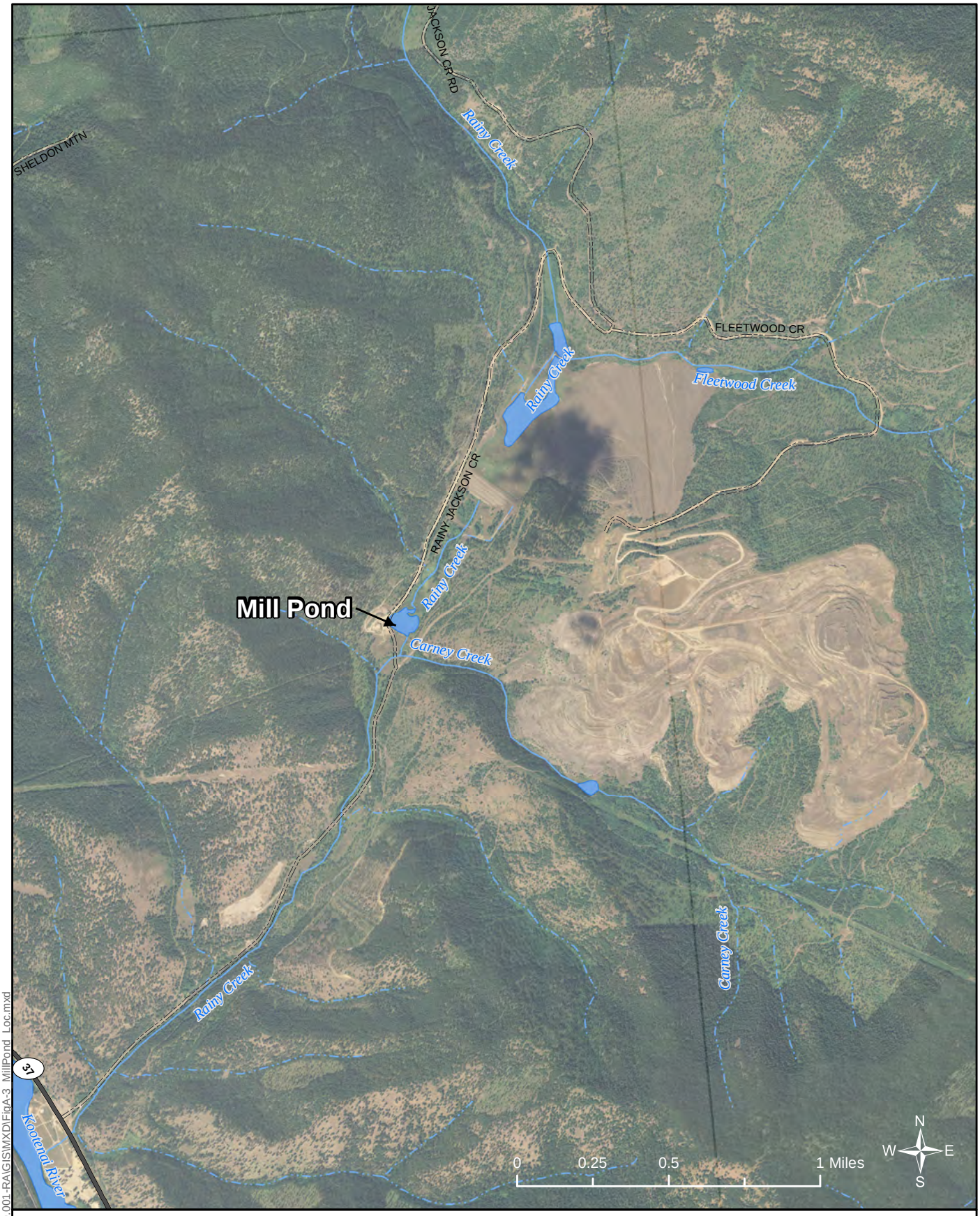

CDM Smith

County Road
Primary Road
Perennial Stream
Intermittent Stream
Open Water

Figure A-3
Location of the Mill Pond in OU3

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Table D-1 General Evaluation Methods for Assessing Asbestos Data Usability

Data Usability Indicator	General Evaluation Method
Precision	Sampling – Review results for field duplicates to provide information on variability arising from medium heterogeneity and sampling. Analysis – Review results for recounts, repreparations, and filter replicates (laboratory duplicates) to provide information on variability arising from analysis methods. Review results for inter-laboratory analyses to provide information on variability and potential bias between laboratories.
Accuracy/Bias	Review results for field blanks, laboratory blanks, and equipment rinsates. Calculate the background filter loading rate and use results to assign detect/non-detect in basic accordance with ASTM 6620-00.
Representativeness	Review relevant field audit report findings and any field/laboratory ROMs for potential data quality issues.
Comparability	Compare the sample collection SOPs, preparation techniques, and analysis methods to previous investigations.
Completeness	Determine the percent of samples that were able to be successfully collected and analyzed (e.g., 99 of 100 samples, 99%).
Sensitivity	Determine if analyses achieved the stopping rules (i.e., the maximum number of LA structures were observed or the maximum filter area was examined).

ASTM = American Society of Testing and Materials
LA = Libby amphibole
QATS = Quality Assurance Technical Support
ROM = record of modification
SAP = sampling and analysis plan
SOP = standard operating procedure
TEM = transmission electron microscopy

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Sampling and Analysis Plan/Quality Assurance Project Plan: Fish and Game Tissue Assessment Libby Asbestos Site, Operable Unit 4

Revision 0 – August 2012

Appendix A
Data Quality Objectives for the Tissue Study

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Appendix A
Data Quality Objectives for the Game Tissue Assessment

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. The design of a study is closely tied to the DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and types of analyses to be performed. The U.S. Environmental Protection Agency (EPA) has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific risk management decisions (EPA 2001a, 2006).

The following sections implement the seven-step DQO process associated with the evaluation of game tissues. DQOs for the evaluation of fish tissues are provided in the *Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP), Operable Unit 3 (OU3), Libby Asbestos Superfund Site – Phase V Part B: 2012 Ecological Investigations* (EPA 2012).

Step 1: State the Problem

Historic mining and milling operations at the mine have resulted in the release of Libby amphibole asbestos (LA) to the environment. Previous investigations of OU3 have demonstrated that LA is present in soils, sediments, surface water, soil, forest duff, and tree bark, with concentrations tending to be highest on the mine site and the forested areas immediately surrounding the mine. Although the exposure pathway of primary concern for humans is inhalation of LA, some studies in animals suggest that ingestion of asbestos fibers can result in the growth of benign intestinal polyps (National Toxicology Program [NTP] 1985). There are several potential scenarios where humans could be exposed to LA via ingestion. One scenario is the ingestion of edible tissues from wild game (deer and elk) that forage in OU3. However, at present, there are no data available on the potential concentrations of LA in edible game tissue from OU3. Thus, data are needed that can be used to determine if human exposures to LA from ingestion of game tissue have the potential to cause unacceptable health risks.

Step 2: Identify the Goal of the Study

The goal of this study is to measure LA concentrations in edible tissues from local game that forage in OU3. These data will be used to evaluate the potential human health risk to humans from the ingestion of LA in the tissues of locally hunted game.

Step 3: Identify the Types of Data Needed

The information inputs that are needed to address the study goal include:

- Reliable measures of LA concentration in edible tissues of deer and elk

- Estimates of the amount of game caught from OU3 that are ingested by hunters and local residents

- Toxicity values by which to assess the potential cancer and non-cancer risks attributable to ingestion of LA

Step 4: Define Study Boundaries

Spatial Bounds

The level of LA in game tissue is expected to be related to the concentration of LA in exposure media and the length of time the game have been exposed to these media (i.e., older game animals are likely to have higher tissue concentrations than younger animals). Thus, to ensure that the evaluation focuses on the high-end of the potential range of exposures, game animals should be collected from a location with high LA concentrations. As noted above, environmental concentrations of LA tend to be highest on the mine site and the forested areas immediately surrounding the mine.

Temporal Bounds

Based on the expectation that the concentration of LA in game tissue will tend to increase over time as a function of the long-term cumulative exposure pattern, it is unlikely that tissue burdens will vary substantially due to short-term fluctuations in media concentrations. Thus, the timing of the game collection is primarily based on ease of sample collection and game permit requirements.

Step 5: Develop the Analytic Approach

The data from this study will be used to estimate the high-end intake and risk to people from ingestion of LA-contaminated game.

Intake

The average daily intake of LA structures due to ingestion of game will be calculated as follows:

$$\text{Intake} = C_{game} \cdot IR \cdot F_{game}$$

where:

Intake = Long-term average daily intake of LA structures from ingestion of game (structures per day [s/day])

C_{game} = Concentration of LA in game tissue (LA structures per gram, wet weight [s/g, ww])

IR = Long-term average ingestion rate of game (grams per day, wet weight [g, ww/day])

F_{game} = Fraction of game ingested that is from animals caught locally (within the Libby Valley)

Risk Evaluation

Data on the health risks from ingestion of asbestos are limited. Studies in animals performed by the NTP in the mid-1980s indicated that ingestion of chrysotile asbestos that contained mainly fibers longer than 10 micrometers (μm) caused an increase in the incidence of benign intestinal polyps in male rats (NTP 1985). This effect was not statistically significant in female rats, or in rats exposed to short chrysotile or to amphibole asbestos.

The EPA has determined that the occurrence of the benign polyps in male rats, combined with other available lines of evidence, provides limited evidence of carcinogenicity for ingested asbestos (EPA 1988). Based on this, EPA (1988) derived a 95% upper confidence limit on the potency of fibers longer than 10 μm to be 1.4E-13 $(s/L)^{-1}$. This potency value was then used to derive a drinking water maximum contaminant level (MCL) of 7 million structures longer than 10 μm per liter (7E+06 s/L) (54 Fed. Reg. 22062 1989; 56 Fed. Reg. 3526 1991). This MCL is a 95% upper confidence limit on the concentration of asbestos fibers longer than 10 μm that may cause a 1E-06 cancer risk of intestinal neoplasia and possibly cancer. Assuming a drinking water intake of 2 liters per day (L/day) (the same intake rate as used to derive the MCL), the potency factor, expressed as risk per fiber per day, is computed as follows:

$$\text{Potency factor} = 1.4E\text{-}13\ (s/L)^{-1} / (2\ L/day) = 7.0E\text{-}14\ (s/day)^{-1}$$

Assuming this value is applicable to LA, the upper-bound on the risk of neoplasia and potentially cancer in people who ingest LA-contaminated tissues is then calculated as follows:

$$\text{Excess Risk} = \text{Intake}\ (s/day) \cdot \text{Potency factor}\ (s/day)^{-1}$$

Data Use and Interpretation

Risk estimates derived as described above will provide a basis for the EPA to determine, in consultation with MDEQ, whether game tissue ingestion is a pathway of potential concern for local hunters. The EPA guidance contained in Office of Solid Waste and Emergency Response Directive 9355.0-30, *Role of the Baseline Risk Assessment in Superfund Remedy Selection Decisions*

(EPA 2001b), indicates that if the cancer risk to an individual does not exceed 1E-04, then remedial action is generally not warranted unless there are adverse environmental affects.

Risks will be calculated using the measured game tissue LA concentrations, assuming a game intake rate that is typical for residents of Libby. If risks based on the tissue concentrations observed in game do not exceed a level of concern, then it will be concluded that risks from LA from ingestion of local game are acceptable. If the results of this study yield a cancer risk that exceeds 1E-04, then it will be concluded that long-term ingestion of game from the Site may pose unacceptable heath concerns.

Step 6: Specify Acceptance Criteria

In evaluating the results of any study, two types of decision errors are possible:

- A *false negative decision error* occurs when it is decided that ingestion of game tissues is safe when it is not safe.

- A *false positive decision error* occurs when it is decided that ingestion of game tissues is not safe when it is safe.

The EPA is most concerned about guarding against the occurrence of false negative decision errors, since an error of this type may leave humans exposed to unacceptable levels of LA in game tissue. To minimize the chances of underestimating the true amount of exposure and risk, the EPA generally recommends that risk calculations be based on the 95% upper confidence limit (95UCL) of the sample mean (EPA 1992). However, applications currently utilized to calculate 95UCLs (i.e., ProUCL) are not designed for asbestos data sets and are not recommended for use (EPA 2008). The EPA is presently working to develop a new software application that will be appropriate for use with asbestos data sets, but the application is not yet available for use. Because the 95UCL cannot presently be calculated with confidence, risk calculations will be based on the sample mean, only, as recommended by EPA (2008). This means that risk estimates may be either higher or lower than true values, and this will be identified as a source of uncertainty in the risk assessment.

The EPA is also concerned with the probability of making false positive decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources. Because it is not possible at present to quantify the uncertainty in the mean of an asbestos data set as a function of the number of samples, it is not possible to specify a minimum number of samples required to minimize the risk of false positive decision errors. In addition, the number of game animals that can be sacrificed is limited, thus it may not be possible to collect enough samples to minimize the potential for making false positive decision errors.

Step 7: Optimize the Study Design

Based on the DQOs presented above, the following are key study design features of the game tissue burden assessment.

Game Species

Based on observations of game species at the Site and information on game consumption for the Libby area, the deer (either mule deer, *Odocoileus hemionus*, or white-tailed deer, *Odocoileus virginianus*) and elk (*Cervus elaphus*) are the target game species for collection. Game species are expected to be exposed LA primarily as a result of feeding behaviors. Deer and elk tend to forage in upland areas and both species have been observed in OU3.

Age and Sex

It is expected that that the concentration of LA in game tissue will tend to increase over time as a function of the long-term cumulative exposure pattern (age). Thus, the preference is to collect deer and elk that are older. In general, body size and antler size can help indicate the age of the deer or elk, but these may be misleading.

To the extent feasible, preference will be given to the collection of female deer and elk as the numbers of female animals are expected to be greater compared to males and body size is likely to be smaller and more manageable to transport.

As it is important to understand the age of the animals in relation to the tissue burdens, the jawbones and teeth will be collected and examined to provide information on animal age.

Target Tissues

For assessing potential human health risks associated with the ingestion of LA in deer and elk tissue, flank muscle samples will be collected. To assess potential tissue burdens in other organs, tissues will also be collected from other organs, including the heart, liver, lung, kidney, and diaphragm.

References

EPA. 1988. *Drinking Water Criteria Document for Asbestos (Draft)*. U.S. Environmental Protection Agency, Washington, D.C.

_____. 2001a. *EPA Requirements for Quality Assurance Project Plans – EPA QA/R-5*. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-01/003. March. http://www.epa.gov/quality/qs-docs/r5-final.pdf

_____. 2001b. *Role of the Baseline Risk Assessment in Superfund Remedy Selection Decisions.* Office of Solid Waste and Emergency Response Directive 9355.0-30.

_____. 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4*. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/240/B-06/001. February. http://www.epa.gov/quality/qs-docs/g4-final.pdf

_____. 2008. *Framework for Investigating Asbestos-Contaminated Sites. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Asbestos Committee of the Technical Review Workgroup*. OSWER Directive #9200.00-68. http://www.epa.gov/superfund/health/contaminants/asbestos/pdfs/framework_asbestos_guidance.pdf

Foresman, K.R. 2001. *The Wild Mammals of Montana*. Special Publication 12. The American Society of Mammalogists. 278 pp.

Jensen, W. 1999. Aging Elk. *North Dakota Outdoors* 62(2):16-20. Jamestown, ND: Northern Prairie Wildlife Research Center Online. http://www.npwrc.usgs.gov/resource/mammals/agelk/index.htm

Jensen, Bill. 2001. Aging Moose. North Dakota Outdoors 64(4):17-20. Jamestown, ND: Northern Prairie Wildlife Research Center Online. http://www.npwrc.usgs.gov/resource/mammals/mooseage/index.htm (Version 27AUG2002).

NTP (National Toxicology Program). 1985. Technical report series no. 295. *Toxicology and carcinogenesis studies of chrysotile asbestos (CAS No. 12001-29-5) in F344/N rats (feed studies)*. Research Triangle Park, NC: U.S. Department of Health and Human Services, Public Health Service, National Institutes of Health. NIH Publication No. 86-2551.

Sampling and Analysis Plan/Quality Assurance Project Plan: Fish and Game Tissue Assessment Libby Asbestos Site, Operable Unit 4

Revision 0 – August 2012

Appendix B
Standard Operating Procedures (SOPs)

SOP ID	SOP Description
Field Procedures	
EPA-LIBBY-2012-01	Field Logbook Content and Control
EPA-LIBBY-2012-02	Photographic Documentation of Field Activities
EPA-LIBBY-2012-04	Field Equipment Decontamination
EPA-LIBBY-2012-05	Handling Investigation-Derived Waste
EPA-LIBBY-2012-06	Sample Custody
EPA-LIBBY-2012-07	Packaging and Shipping of Environmental Samples
CDM-LIBBY-09	GPS Coordinate Collection and Handling
EPA-LIBBY-2012-14	Filleting Fish Samples
EPA-LIBBY-2012-15	Gross Necropsy and Tissue Sample Collection for Game Animals
Sample Preparation and Analysis Procedures	
EPA-LIBBY-2012-13	Analysis of Tissue for Asbestos
Data Review Procedures	
EPA-LIBBY-09	TEM Data Review and Data Entry Verification
EPA-LIBBY-11	FSDS Data Review and Data Entry Verification

***The most recent versions of all field SOPs are provided electronically in the Libby Field eRoom (https://team.cdm.com/eRoom/R8-RAC/Libby).*

***The most recent versions of all laboratory and data review SOPs are provided electronically in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab).*

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Sampling and Analysis Plan/Quality Assurance Project Plan: Fish and Game Tissue Assessment Libby Asbestos Site, Operable Unit 4

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Appendix C
Montana Fish, Wildlife, and Parks Permit

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MONTANA DEPARTMENT OF FISH, WILDLIFE and PARKS

Wildlife Bureau

P. O. Box 200701 · Helena, MT 59620-0701 · (406) 444-2612

SCIENTIFIC COLLECTOR'S PERMIT

Permit # 2012-073

IACUC # NA

Federal Permit #: NA ()

Permitted Activities: Scientific Collection

Fee: No Fee

Date Issued: 8/1/2012

Date Expires: 12/31/2012

One Year Permit only (see note in conditions)

Permit issued to:	Liz Fagen
Address:	108 E 9th St. Libby, MT 59923
Phone number:	406-293-6194
Email:	fagen.elizabeth@epa.gov
Associated with:	Environmental Protection Agency

A report of activities conducted under the provisions of this permit must be sent to Montana Fish, Wildlife and Parks, Attn: Wildlife Bureau, POB 200701, Helena, MT 59620-0701 by December 31 annually. The report should list the number of animals handled, including species, date, location (GPS location in UTM coordinates or latitude-longitude if possible; or the legal description in Township, Range, Section and quarter section; otherwise a detailed description of location), other known biological information (sex, age, etc.) and/or cause of death if known. This information will be used for administrative purposes, and to supplement location information housed in the Montana Natural Heritage Program on species.

Subpermittees:

Mike Cirian , David Berry , Deb McKean , Victor Ketellapper , Nathan Smith

Copies of this permit must be in possession while engaged in activities.

This permit is not transferable.

Montana Fish, Wildlife & Parks

Ken McDonald
Bureau Chief, Wildlife Bureau

Montana Fish, Wildlife & Parks

CC:

MONTANA DEPARTMENT OF FISH, WILDLIFE and PARKS

Wildlife Bureau

P. O. Box 200701 · Helena, MT 59620-0701 · (406) 444-2612

SCIENTIFIC COLLECTOR'S PERMIT

Permit # 2012-073

Permit Conditions:

Authorized to lethally remove one adult elk (either sex) and one adult deer (either sex) OR two adult deer (either sex) within the private property owned by WR Grace at the Libby Vermiculite Mine for the purposes of toxicology testing. The animals will be killed by Mike Cirian as a subpermittee of this permit. Desired harvest locations may be baited with grains, corn or commercially available bait products. Bait must be removed immediately following the harvest of desired sample animals or after any bear activity is detected at the site.

Any meat found fit for human consumption after testing will be donated to the local food bank.

Note: This permit expires upon the date listed above. A new application form must be submitted in order to continue this project or associated work beyond the expiration date.

Other Relevant Montana Code Annotated and Administrative Rules of Montana:

MCA 87.2.806- MCA 87.5.109- MCA 87.1.221-

Copies of this permit must be in possession while engaged in activities.

This permit is not transferable.

Montana Fish, Wildlife & Parks

CC:

Sampling and Analysis Plan/Quality Assurance Project Plan: Fish and Game Tissue Assessment Libby Asbestos Site, Operable Unit 4

Revision 0 – August 2012

Appendix D
Analytical Requirements Summary Sheet
[TISSOU4-0812]

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SAP/QAPP REQUIREMENTS SUMMARY #TISSOU4-0812
SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS FOR ASBESTOS

Title: Sampling and Analysis Plan/Quality Assurance Project Plan, Fish & Game Tissue Assessment, Libby Asbestos Superfund Site, Operable Unit 4

SAP Date (Revision): August 2012 (Revision 0)

EPA Technical Advisor: Elizabeth Fagen (303-312-6095, Fagen.Elizabeth@epa.gov)
(contact to advise on DQOs of SAP related to preparation/analytical requirements)

Sampling Program Overview: This program will include the collection of two game animals (elk, deer) from OU3 and the analysis of muscle and organ tissue samples for asbestos by TEM. In addition, this program includes the preparation (filleting) and analysis of fish samples collected from the Mill Pond for asbestos by TEM.

Sample ID Prefix: TS-_ _ _ _ _

Estimated number and timing of field samples:
Fish tissue samples will be provided in September 2012; game tissue samples will be collected in October-November 2012 timeframe (exact dates will depend upon when animals can be harvested).

>> Fish Tissue = 12 samples (fillet)
>> Game Tissue = 16 samples (muscle and organ)

TEM Preparation and Analytical Requirements for Tissue Samples:

Medium Code	Medium, Sample Type	Preparation Details [a]				Analysis Details			Applicable Laboratory Modifications (current version of)
		Investi-gative?	Indirect Prep?		Filter Archive?	Method	Recording Rules [c]	Analytical Sensitivity/Prioritized Stopping Rules	
			With Ashing [b]	Without Ashing					
A	Tissue	Yes	Yes (see SOP EPA-LIBBY-2012-13 for sample preparation details)	No	Yes	TEM – ISO 10312	All asbestos; L: > 5 µm W: ≥ 0.25 µm AR: ≥ 3:1	Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) 25 PCME LA structures are recorded ii) 0.25 mm^2 of filter has been examined (approx. 25 grid openings)	LB-000016, LB-000029, LB-000066D, LB-000067, LB-000085

(a) Fish will be sent as whole body samples; the analytical laboratory is responsible for filleting each fish in accordance with SOP EPA-LIBBY-2012-14. One fillet will be collected and analyzed from each fish. In addition, for one fish, the remaining fillet should be analyzed separately as a field duplicate (the chain-of-custody will indicate which fish should be used for the field duplicate).
(b) A total of 3 replicate filters will be generated for each tissue sample (i.e., 3 different aliquots of the ash residue will be used to create 3 replicate filters).
(c) If observed, chrysotile and other amphibole asbestos should be recorded.

TEM Preparation and Analytical Requirements for Equipment Rinsate Water Samples:

Medium Code	Medium	Preparation Details[(d)]				Analysis Details			Applicable Laboratory Modifications (current version of)
		Investi-gative?	Indirect Prep?		Filter Archive?	Method	Recording Rules	Analytical Sensitivity/ Stopping Rules	
			With Ashing	Without Ashing					
E	Rinsate Water	Yes	No	No	Yes	Standard TEM; ISO 10312	All asbestos [(e)]; L: $\geq$ 0.5 µm AR: $\geq$ 3:1	Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) sensitivity of 50,000 L^{-1} is achieved ii) 25 structures are recorded iii) A total filter area of 1.0 mm^2 has been examined (approx. 100 grid openings)	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085

(d) Sample and filter preparation should be performed in basic accordance with EPA Method 100.2 (as modified by LB-000020A). Grid preparation should be performed in basic accordance with Section 9.3 of ISO 10312:1995(E).
(e) If observed, chrysotile structures should be recorded, but chrysotile structure counting may stop after 25 structures have been recorded.

Analytical Laboratory Quality Control Sample Frequencies:

TEM [(f)]:
- Lab Blank – 4%
- Recount Same – 1%
- Recount Different – 2.5%
- Verified Analysis – 1%
- Interlab – 0.5%
- Repreparation – 1%

Addtl TEM, for tissue:
- Drying Blank – 1 per batch
- Filtration Blank – 2%

(f) See LB-000029 for selection procedure and QC acceptance criteria

Requirements Revision:

Revision #:	Effective Date:	Revision Description
0	8/31/2012	N/A

Asbestos Analytical Laboratory Review Sign-off:

☐ EMSL – Cinnaminson [sign & date:_____] ☐ ESAT [sign & date:_____]

[Checking the box and signing (electronically) above indicates that the laboratory has reviewed and acknowledged the preparation and analytical requirements associated with the specified SAP.]