

Summer 2016

EVALUATION OF LIBBY AMPHIBOLE ASBESTOS IN THREE MEDIA SOURCES VIA POLARIZED LIGHT MICROSCOPY AND TRANSMISSION ELECTRON MICROSCOPY

Kalli McCloskey

Montana Tech of the University of Montana

Follow this and additional works at: http://digitalcommons.mtech.edu/grad_rsch



Part of the [Occupational Health and Industrial Hygiene Commons](#)

Recommended Citation

McCloskey, Kalli, "EVALUATION OF LIBBY AMPHIBOLE ASBESTOS IN THREE MEDIA SOURCES VIA POLARIZED LIGHT MICROSCOPY AND TRANSMISSION ELECTRON MICROSCOPY" (2016). *Graduate Theses & Non-Theses*. 98.
http://digitalcommons.mtech.edu/grad_rsch/98

This Thesis is brought to you for free and open access by the Student Scholarship at Digital Commons @ Montana Tech. It has been accepted for inclusion in Graduate Theses & Non-Theses by an authorized administrator of Digital Commons @ Montana Tech. For more information, please contact sjuskiewicz@mtech.edu.

EVALUATION OF LIBBY AMPHIBOLE ASBESTOS
IN THREE MEDIA SOURCES VIA POLARIZED LIGHT MICROSCOPY
AND TRANSMISSION ELECTRON MICROSCOPY

by
Kalli McCloskey

A thesis submitted in partial fulfillment of the
requirements for the degree of

Industrial Hygiene

Montana Tech

2016



Abstract

Studies have shown that the town of Libby, Montana and surrounding forested areas have been contaminated with Libby amphibole asbestos (LA) by activity from the vermiculite mine located northeast of Libby. The mine was in production from the 1920s until its closing in 1990. The United States Environmental Protection Agency (USEPA) created the operable units (OU) within the superfund site due to the LA contamination. In addition to the work completed in this research, previous studies have shown that there is an exposure pathway for the United States Department of Agriculture, Forest Service (USFS) personnel tasked with forest maintenance in and around the Tubb gulch OU3 area.

Tree bark, duff, and soil composite samples were collected to characterize the LA contamination in the Tubb Gulch area west of the mine. Composite samples were analyzed first by polarized light microscopy (PLM) (for bark and duff) and second by transmission electron microscopy for all media sources (TEM). The objective was to determine if PLM analysis could be used as a primary analytical tool for bark and duff sampling, since TEM is considerably more expensive and takes longer for the laboratory analysis.

Fifteen positive composite samples that were initially analyzed via PLM were selected for further analyses via TEM for bark and duff media types. Bark sampling results revealed positive TEM values in 14 of 15 (93%) of the samples and duff sampling results revealed positive TEM values in 15 of 15 (100%) of the samples. These data suggest that PLM analysis may be a reliable initial analytical screening method. Since the LA concentrations for bark were expressed as fibers per surface area of bark and the LA concentrations for duff were expressed as fibers per gram, statistical correlation analyses could not be performed. A further limitation with this assessment is that bark and duff samples revealing non-detect values via PLM analysis were not selected for TEM analysis; therefore, the potential for false negative results via PLM was not assessed.

The results of this study were valuable in further characterizing the Tubb Gulch area. All bark and duff composite samples revealed the presence of asbestos structures via TEM with the exception of one bark sample. Based on these source media results, there is a potential for LA exposure to USFS personnel or members of the public when working or recreating in the Tubb Gulch area. It is important to note that the results did not follow a concentration gradient in that some of the lowest concentrations were detected closest to the mine. These data suggest that LA contamination was dispersed not only from the mining activities but during transportation of the vermiculite concentrate to Libby and to the train loading facility.

Keywords: Libby Amphibole Asbestos, Asbestos, Libby Montana, Transmission Electron Microscopy, Polarized Light Microscopy

Dedication

I wish to thank my husband Mike for all his support. I would also like to thank my parents for pushing me and helping me throughout my life, with support on my paper, and college career.

Acknowledgements

I would like to start with acknowledging Julie Hart and Terry Spear for getting the internship with Portage Inc. and for all her help with my thesis. I would like to acknowledge Portage Inc., Pat Seccomb, for the funding and internship opportunity in Libby, MT. Also Brienne Meyer for supervising, training, and decontamination in the field.

Table of Contents

ABSTRACT	II
DEDICATION	III
ACKNOWLEDGEMENTS	IV
TABLE OF CONTENTS.....	V
LIST OF TABLES	VII
LIST OF FIGURES.....	VIII
GLOSSARY OF TERMS.....	IX
 1. INTRODUCTION	 1
2. HISTORY	3
3. VERMICULITE CHARACTERISTICS.....	5
4. TOXICOLOGY OF LIBBY AMPHIBOLE.....	6
5. EPIDEMIOLOGY.....	7
5.1. <i>Cancer Effects</i>	7
5.2. <i>Non-Cancer Effects</i>	8
5.2.1. Reference Concentration (RfC).....	9
6. PREVIOUS LIBBY AMPHIBOLE EXPOSURE STUDIES.....	11
7. RESEARCH OBJECTIVE	13
8. BULK ASBESTOS SAMPLING ANALYTICAL METHODS	16
8.1. <i>Polarized Light Microscopy</i>	16
8.1.1. PLM Complications.....	16
8.2. <i>Transmission Electron Microscopy</i>	17
8.2.1. TEM Complications.....	18
9. METHODS	19

9.1.	<i>Sampling Plan</i>	19
9.1.1.	Bark Samples	25
9.1.2.	Duff Samples	26
9.1.3.	Soil Samples.....	26
9.1.4.	Field Duplicate Samples	26
9.2.	<i>PLM Analytical Method</i>	26
9.2.1.	PLM Sample Pre-Preparation	27
9.2.2.	PLM Sample Preparation	28
9.3.	<i>TEM Analytical Method</i>	28
10.	HYPOTHESES TESTS	30
11.	HYPOTHESES.....	31
11.1.	<i>Bark</i>	31
11.2.	<i>Duff</i>	31
11.3.	<i>Relationship between Media Sources</i>	31
12.	RESULTS AND DISCUSSION	32
12.1.	<i>Bark</i>	33
12.2.	<i>Duff</i>	35
12.3.	<i>Soil</i>	44
13.	CONCLUSION	45
	REFERENCES CITED	47
	APPENDIX A:	52
	PLM RESULTS: DUFF	52
	PLM RESULTS: BARK	54
	PLM RESULTS: SOIL	56

List of Tables

Table I: Sampling Plan.....	19
Table II: Bark PLM and TEM Analysis Results.....	33
Table III: Duff PLM and TEM Analysis Results.....	35
Table IV: Matrix Scoring System for Bark and Duff Concentrations	38
Table V: Matrix Scores for Bark and Duff Concentration Results.....	38
Table VI: Soil PLM Analysis Results.....	44

List of Figures

Figure 1: USEPA's Libby Amphibole Asbestos National Priorities List Superfund Sites Operable Units (Hestmark, 2015) Bottom right corner WindRose Diagram for Zonolite Mountain: displays wind speed and direction (EPA, 2013c)	15
Figure 2: TEM photos of LA form EMSL Analytical Inc lab (left) at 720 magnification (right) at 20,000 magnification	18
Figure 3: Tubb Gulch Sampling Areas	21
Figure 4: Tubb Gulch North Sampling Area	22
Figure 5: Tubb Gulch Central Sampling Area	23
Figure 6: Tubb Gulch South Sampling Area	24
Figure 7: Bark Samples TEM Analysis Concentration Results	36
Figure 8: Duff Samples TEM Analysis Concentration Results	37
Figure 9: Matrix Scoring System for Bark and Duff Comparison	39
Figure 10: Bark Topography Matrix Rankings	42
Figure 11: Duff Topography Matrix Rankings	43

Glossary of Terms

Term	Definition
$\mu\text{g}/\text{m}^3$	Microgram per meter cubed
ANOVA	Analysis of Variance
ATSDR	Agency for Toxic Substance and Disease Registry
CH	Chrysotile Fibers
EDS	Energy Dispersive Spectroscopy
EMSL	EMSL Analytical Inc
fiber/cc	Fiber/cubic centimeter of air
fibers/ m^3	Fibers per cubic meter of air
GPS	Global Positioning System
H_i	Alternative Hypothesis
H_o	Null Hypothesis
IUR	Inhalation Unit Risk
LA	Libby Amphibole Asbestos
LOAEL	Lowest-observed-adverse-effect level
LOD	Level of Detection
NAM	Non-asbestos Material
ND	Nondetect
NIOSH	National Institute for Occupational Safety and Health
NOAEL	No-observed-adverse-effect level
NPL	National Priorities List
OA	Other Amphibole-type Asbestos
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PCME	Phase Contrast Microscopy Equivalent
PEL	Permissible Exposure Limit
PHA	Public Health Assessment
PLM	Polarized Light Microscopy
PLM-Grav	Polarized Light Microscopy-Gravimetric
PLM-VE	Polarized Light Microscopy-Visual Estimation
REL	Recommended Exposure Limit
RfC	Reference Concentration
RfD	Oral Reference Dose
SAED	Selected Area Electron Diffraction
TEM	Transmission Electron Microscopy
TR	Traces
TWA	Time-weighted Average
UML	University of Montana- Ward Laboratory
USEPA	United States Environmental Protection Agency
USFS	United States Department of Agriculture, Forest Service

1. Introduction

In 2005, the presence of Libby amphibole asbestos (LA) was discovered in operable unit 3 (OU) timber stands (Ward et al., 2006). It was postulated that airborne LA had been dispersed from the former vermiculite mine and had impacted or was intercepted on the surface of tree bark. Further human LA exposure studies (Hart et al., 2007, Hart et al., 2009; Ward et al., 2009; Ward et al., 2012) suggest that there is an inhalation exposure pathway for United States Department of Agriculture, Forest Service (USFS) personnel tasked with firefighting and timber and vegetation management in this area and potentially in the neighboring Tubb Gulch area. In addition, wildfires pose a threat in this area, which may cause the LA to become airborne in the smoke plane, potentially spreading LA further across the town of Libby and surrounding forested areas.

The USFS identified a need to understand the extent of LA in Tubb Gulch and its potential health risks to USFS personnel and as a result, they funded an extensive sampling effort. However, the concentration and distribution of LA within the Tubb Gulch timber stands were not characterized sufficiently before the data presented in this thesis. Sampling was conducted in order to characterize the extent of LA contamination present in Tubb Gulch's source media. The source media includes tree bark, duff, and soil. Duff is defined as the biomass accumulated on the forest floor and consists mainly of shed vegetation such as leaves, branches, bark and stems existing in various stages of decomposition above the soil surface (Lemieux, 2012).

In February 2016, the U.S. Environmental Protection Agency (USEPA) selected a final cleanup plan for the remaining portions of Libby, Montana USEPA's Superfund Site with an

exception of the former Libby vermiculite mine and the bordering forested area (EPA, 2016).

The USEPA does not have a cleanup plan for the forested area because there is not presently sufficient data to characterize the hazards to human health and the environment. The data in this thesis assists in characterizing the extent of LA contamination, specifically in Tubb Gulch. This area is located east of the Libby vermiculite mine in the vicinity of USEPA's Superfund Site Operable Unit 3 (OU).

Source media was analyzed by two separate methods, polarized light microscopy (PLM) and transmission electron microscopy (TEM). Data analysis considering these two analytical methods was used to determine the detection accuracy specific to LA of the three source media. Interpretation of the data will support the USFS's decision to utilize PLM and/or TEM analysis for an approved detection method to define the extent of LA concentration in Tubb Gulch.

2. History

Libby is a small town of about 2,600 residents and is located in Lincoln County, in northwest Montana. The largest known deposit of vermiculite in the world is approximately seven miles northeast of Libby, Montana. The vermiculite mine was one of the main employers for the residents of Libby from 1924 to 1990 and was owned and operated by the Zonolite Corporation until 1963. The mine was then purchased and operated by W.R. Grace until the closing in 1990 (ATSDR, 2010).

Vermiculite from the mine is naturally contaminated with a toxic and highly friable form of asbestos referred to as LA. Libby amphibole was originally reported as tremolite, but was later identified as 84% wickite, 11% richterite, and 6% tremolite (Meeker, 2003). Mining, milling, and processing of vermiculite at the site were known to have attributed to releases of Libby amphibole asbestos into the environment. This release of LA caused an inhalation hazard known to increase the risk of cancer and non-cancer effects in the lungs of humans (EPA, 2007, 2013).

Libby, Montana was proposed for the National Priorities List (NPL) on February 26, 2002 and listed on October 24, 2002. The Agency for Toxic Substances and Disease Registry (ATSDR) is required by congress to conduct public health assessments (PHA) on all sites proposed for the NPL. In the PHA, ATSDR evaluated the public health implications of the defined OU sites using available environmental data, potential exposure scenarios, community health concerns, and health outcome data (ATSDR, 2003).

The ATSDR concluded from the PHA that: (a) people in the Libby area were exposed to hazardous levels of LA in the past; (b) people in the Libby area have elevated levels of disease and death associated with exposure to LA; (c) people could still be exposed to hazardous levels of LA near current source areas. These levels could be especially hazardous to sensitive

populations, including people who have been exposed for many years already, smokers, and young children; and (d) the exact level of risk associated with low-level exposure to asbestos cannot be determined due to uncertainties in the analysis and toxicology of Libby asbestos. Continuing exposures to LA pose an unacceptable risk to residents and workers who have already been exposed for many years (ATSDR, 2003).

3. Vermiculite Characteristics

While the vermiculite mine was in production, it supplied up to 80% of the world's supply of vermiculite (ATSDR, 2010). Vermiculite is a unique mineral with the ability to exfoliate, or expand, upon heating. Exfoliated vermiculite has many different commercial uses such as inclusion in concrete aggregates, loose-fill insulation, horticulture applications such as soil conditioning, and a bulk carrier for agriculture chemicals (ATSDR, 2010).

Libby amphibole fibers do not have any detectable odor or taste, do not dissolve in water or evaporate into the air, are resistant to heat, fire, chemical, and biological degradation and can remain virtually unchanged in the environment for a long period of time. In consequence, individual fibers can be suspended into the air creating a hazardous environment and can potentially be inhaled and could cause chronic respirable and upper gastrointestinal health effects (ATSDR, 2008).

4. Toxicology of Libby Amphibole

People may be exposed to LA by two exposure routes: inhalation and ingestion. Of these two exposure routes, inhalation exposure of LA is considered to be the greatest concern (EPA, 2014). Although oral ingestion is an exposure route, health risks from ingestion are low compared to health risks from inhalation (ATSDR, 2008).

Libby amphibole asbestos fibers in vermiculite are typically not inherently hazardous unless the asbestos is released from the source into air where it can be inhaled. The LA fibers may become airborne in a number of ways such as natural forces, wind blowing over contaminated soil, or human activities that disturb contaminated sources, such as soil or indoor dust (EPA, 2014).

According to Meeker (2003), the average diameters of respirable LA fibers are between $0.56 \pm 0.45\mu\text{m}$. The aerodynamic properties of fibers are such that fibers as long as $50\mu\text{m}$ or as big as $3\mu\text{m}$ in diameter can reach the alveolar region and are considered respirable (Hinds, 1999). Once lodged in an alveolus, large fibers cannot be removed by normal clearance mechanisms. They are insoluble in lung fluids, too long to be engulfed by macrophages, and only slightly able to migrate to lymph nodes. Macrophages cannot engulf long fibers so they release cytosine proteins that initiate the enzymes that cause fibrosis (the scarring and thickening of the alveolar surfaces associated with asbestosis). Shorter asbestos fibers less than $5\mu\text{m}$ in length can be cleared by the normal clearance mechanisms. Each of the asbestos-related diseases were associated with fibers in a specific size range (Hinds, 1999).

5. Epidemiology

Libby vermiculite workers had experienced significant excess deaths from all causes: lung cancer, mesothelioma, and asbestosis. Mortality from asbestosis and lung cancer increased with increasing cumulative exposure to airborne LA. Although the mine has ceased operations, historic or continuing releases of LA from mine-related materials could be serving as a source of ongoing exposure and risk to current and future residents and workers in the area (Sullivan, et al. 2007).

5.1. Cancer Effects

Epidemiological studies have reported increased mortality from cancer in workers exposed to asbestos (EPA, 2014). Mesothelioma is the most common reported cancer resulting from exposure to LA. Mesothelioma is a cancer of the membrane (pleura) that encases the lungs and lines the chest and peritoneal cavity. This cancer can spread to tissues surrounding the lungs or other organs (ATSDR, 2008).

Lung cancer is also caused by LA and is cancer of the lung tissue specifically bronchogenic carcinoma. The combination of tobacco smoking and LA exposure greatly increases the risk of developing lung cancer. Laryngeal cancer, cancer of the larynx (voice box). Additionally, some evidence suggests that acute oral exposure can induce precursor lesions of colon cancer and chronic oral exposure can lead to an increased risk of gastrointestinal tumors (ATSDR, 2008).

Cancer risks are defined by an inhalation unit risk (IUR). Typically, an IUR is defined as a possible upper bound on the estimate of cancer risk per microgram per meter cubed ($\mu\text{g}/\text{m}^3$) of air breathed for 70 years. But for LA fibers exposure, the IUR is expressed as cancer risk per fiber per cubic centimeter (fiber/cc). For LA the IUR represents the lifetime risk of mortality

from either mesothelioma or lung cancer in the general US population from chronic inhalation exposure to LA at a concentration of 1 fiber/cc of air (EPA, 2011).

EPA considers cumulative excess cancer risks that are below $1\text{E-}06$ to be insignificant, and risks above $1\text{E-}04$ to be sufficiently large that some form of remedial action is desirable (EPA, 2014). A concentration of $1\text{E-}03$ is equal to 0.001 fibers/cc, and a concentration of $1\text{E-}04$ is equal to 0.0001 fibers/cc (EPA, 2013b)

5.2. Non-Cancer Effects

Noncancerous toxicity refers to adverse health effects or toxic endpoints, other than cancer and gene mutations, that are due to the effects of environmental agents on the structure or function of various organs systems. Most chemicals that produce noncancerous toxicity do not cause a similar degree of toxicity in all organs, but usually demonstrate major toxicity to one or two organs, referred to as target organs (EPA, 1994).

Non-cancer effects from asbestos exposure include asbestosis (formation of scar tissue in the lung parenchyma) and pleura plaques (localized areas of thickening of pleura); diffuse pleural thickening, extensive, non-discrete thickening of pleura; pleural calcification, calcium deposition in pleural areas thickened from chronic inflammation and scarring; and pleural effusions (the membrane surrounding the lungs), such as pleural effusions (excess fluid accumulation in the pleural space). (ATSDR, 2008; EPA, 2014).

The latency period for noncancerous respiratory effects is usually 15 to 40 years from the time of initial exposure to LA (ATSDR, 2008). Exposure to LA does not necessarily mean a person will develop asbestos-related health effects. In general, increasing frequency, duration, and intensity of exposure are associated with increased risk of disease. Personal risk factors such

as a history of smoking, history of lung disease, and genetic susceptibility are important determinants of actual risk (ATSDR, 2008).

The National Institute for Occupational Safety and Health (NIOSH) considers asbestos to be an occupational carcinogen and recommends that exposures be reduced to the lowest feasible concentration. For asbestos fibers > 5 microns in length, NIOSH recommends the recommended exposure limit (REL) of 100,000 fibers per cubic meter of air (100,000 fibers/m³), which is equal to 0.1 fibers/cc. As found in 29 CFR 1910.1001, the Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for asbestos fibers is an 8-hr time weighted average (TWA) airborne concentration of 0.1 fiber/cc. Workers could be at risk if exposed to LA in excess of 1 fiber/cc on average over a sampling period of 30min (NIOSH, 2015).

5.2.1. Reference Concentration (RfC)

To determine the risk associated with LA noncancerous health effects, the reference concentration (RfC) is used. The RfC is an estimate of a continuous inhalation exposure of LA to the human population that is likely to be without a substantial risk of harmful noncancerous effects during a lifetime (IRIS, 2014). The RfC considers toxic effects of both the respiratory system and for effects peripheral to the respiratory system. It is expressed in units of fibers/cc. The chronic RfC value is 9×10^{-5} fibers/cc which is substantially lower than the PEL of 1 fiber/cc (EPA, 2014b).

There are no studies in vivo for inhalation routes suitable for derivation of an RfC because they lack adequate exposure-response information and are short-term. Ultimately, the RfC was based on localized pleural thickening derived from a Marysville, OH worker cohort data set. The Marysville cohort was selected because the workers were exposed to lower LA

concentrations relative to Libby cohorts and workers showered and changed at the conclusion of the work shift, resulting in minimal non-occupational exposures (EPA, 2014).

6. Previous Libby Amphibole Exposure Studies

In 2006, researchers from Montana Tech and the University of Montana discovered that trees surrounding the former vermiculite mine served as reservoirs for LA (Ward, 2006). Tree bark and tree core samples were collected at seven locations in OU3 to determine whether LA would be present in bark and if LA was taken up through the root system (core sample). Analysis found that LA fibers were not taken up by the root system, but LA fibers were found in the bark surface samples. It is assumed that the positive bark samples were the result of wind dispersal from the former vermiculite mine activities. The analytical results indicated that the tree bark samples contained between 14 million to 260 million LA fiber/cm² of bark surface area (Ward et al., 2006). This was substantial because, while LA contamination was previously detected in the soils near the former vermiculite mine, tree reservoirs then became an additional media source for potential exposures along with duff.

In 2009, research was conducted to assess potential USFS employee exposures while working near the vermiculite mine, but outside the USEPA restricted zone in OU3 (Hart et al., 2009). Investigators simulated four routine activities: (1) walking through forested areas, (2) conducting tree measurements, (3) constructing a fire line, and (4) performing trail maintenance. Personal breathing zone air samples and clothing wipe samples were collected to assess LA exposure. Samples results were positive for LA, indicating the potential for USFS worker exposure. In addition, this area can be accessed by the general public (Hart et al., 2009).

Combustion trials of LA contaminated wood (Ward et al., 2009) revealed that while the majority of LA fibers remained in the ash, there was also a potential for fibers to be liberated into the ambient air during the combustion process.

In 2012 the USEPA conducted a controlled burn study to provide information about the potential exposure to firefighters for LA fibers in the event of a forest fire, if one should occur in the area around the former mine site in Libby, Montana. A laboratory-scale simulation of a wildfire had to be used because performing a test burn in the field would result in uncontrolled emissions of LA being released into the atmosphere. LA-contaminated duff was used to yield emission factors that could be used to perform exposure assessments that would be based on measured emissions from a combustion environment. Upward motion of air due to convective motion was not able to be simulated in the lab (Lemieux, 2012).

The experiments were performed with the intent to simulate the temperatures encountered in wildfires that may impact the release of LA fibers. Under the conditions that were tested, fractions of phase contrast microscopy equivalent (PCME) asbestos fibers ranging from 88% to 105% appeared to remain behind in the residual bottom ash that remained after the burn was completed for the high temperature burn conditions. Fractions of PCME asbestos fibers ranging from 88% to 115% appear to remain behind in the residual bottom ash that was left after the burn was completed for the Lower Temperature burns. This study suggested that the majority of the LA fibers that are present in the duff do not become entrained into the air emission (Lemieux, 2012). However, this study could not simulate air convection motion which could cause LA fibers to be projected into the air.

7. Research Objective

The Libby, Montana Superfund Site is divided into eight operable units, which encompass both publically and privately owned lands, Figure 1: NPL Libby Superfund Site (Hestmark, 2015). Also in Figure 1 on the right hand corner is a key for a WindRose Diagram which depicts the historical wind direction and speed for the area located in the vicinity of the mine. The WindRose Diagram indicates that the prevailing winds blow towards the north north-east, away from the town of Libby (EPA, 2013c).

As illustrated in Figure 1, Operable Unit 3 mainly consists of property in and around the vermiculite mine and any area impacted by the release and subsequent migration of hazardous substances and pollutants released into the air from the mine (EPA, 2007). The remainder of the outlying forested areas lie within OU4 (residential, commercial, and public) or lie outside the NPL site boundary. Additionally, due to the general wind direction, contamination could potentially have spread into the forested areas from OU1 (the expansion plant) or from transportation of the vermiculite on the highway (OU8).

The objective of this research was to evaluate LA concentrations in bark, duff, and soil via two analytical methods; PLM and TEM and determine whether or not the PLM analysis could be used as the primary analytical method for LA determination. Since the Tubb Gulch timber stands cover approximately 500 acres including some areas of steep terrain, the site offered physical challenges to acquire sufficient data to support USFS management objectives. But the USFS hypothesized that the sampling strategies selected would provide sufficient data to characterize LA concentration and distribution in Tubb Gulch. The sampling conducted by Portage Inc was within Tubb Gulch and includes OU3, part of OU2 and the forested area just

outside the NPL site boundary. The USFS would use the data to assess the potential health and environmental risks associated with LA within Tubb Gulch.

Figure 1: USEPA's Libby Amphibole Asbestos National Priorities List Superfund Sites Operable Units (Hestmark, 2015) Bottom right corner WindRose Diagram for Zonolite Mountain: displays wind speed and direction (EPA, 2013c)

8. Bulk Asbestos Sampling Analytical Methods

8.1. Polarized Light Microscopy

The technique often used for the analysis of bulk asbestos containing material is by polarized light microscopy (PLM). The PLM analysis uses a stereo light microscope to help separate a bulk sample and a polarizing light microscope to identify the fibers among the binders and fillers (Dodson & Hammar, 2011). A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows for the determination of refractive indices relative to specific crystallographic orientations. Morphology and color are also observed while viewing under plane polarized light (Perkins, 1993). Because the size of wavelength of light, PLM analysis is limited to fibers approximately 1 μ m in diameter or thicker (Dodson & Hammar, 2011). The PLM analysis is inexpensive and can be performed on site if a light microscope is available (OSHA 2013).

8.1.1. PLM Complications

The polarized light microscopy initial screening includes ashed fibers, duff, and organic materials ashed in mineralized soils. This will likely result in overestimation of asbestos counted during the initial screening process of PLM.

Although PLM analysis is the primary technique used for bulk asbestos determination, it can show significant bias ending with false negatives and false positives for certain types of materials. This bias could result due to the limitation of PLM detection limits (Dodson & Hammar, 2011). Fibrous and nonfibrous, organic and inorganic components of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Binder and matrix materials may coat fibers, affect color, or obscure optical characteristics to the extent of

masking fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification (Perkins, 1993).

Polarized light microscopy cannot reliably detect asbestos in low concentrations below 1% (10,000 fibers/cc) (EPA, 2014b). Since counting asbestos fibers are limited to structures longer than 5µm and with a defined length-to-width ratio of 3:1 or greater, accountability for fibers smaller than the countable sizes are passed, causing the concentration to be underestimated (Case et al., 2011).

8.2. Transmission Electron Microscopy

Transmission electron microscopy (TEM) analysis has the ability to visualize hundreds of fibers in which PLM was unable to detect. The TEM analysis could provide all three pieces of information required for fiber identification (morphology, chemistry, and structure) (OSHA, 2013). The quantitative working range is 0.04 to 0.5 fiber/cc. The level of detection (LOD) depends on sample volume and quantity of interfering dust, which is <0.01 fiber/cc for atmospheres free of interferences (NIOSH 7402, 1994). Figure 2 shows TEM analysis photos for LA fibers under 720-20,000 magnifications from the EMSL Analytical Inc lab. Transmission Electron Microscopy can be a powerful tool and should be used in conjunction with PLM when necessary to justify PLM analysis results (OSHA 2013).

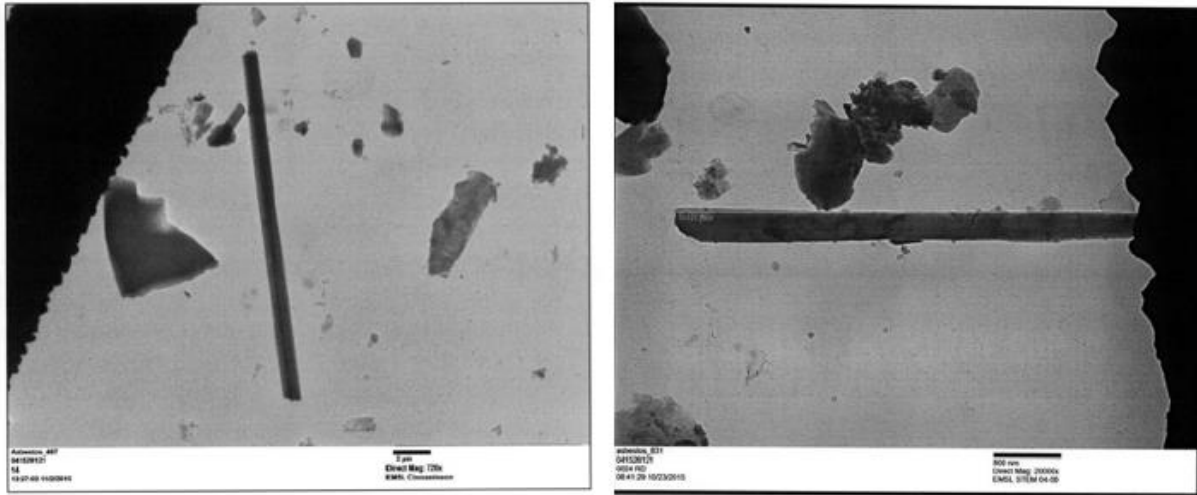


Figure 2: TEM photos of LA form EMSL Analytical Inc lab (left) at 720 magnification (right) at 20,000 magnification

8.2.1. TEM Complications

Elemental composition similar to the LA minerals, may interfere in the TEM analysis. Also some non-amphibole minerals may give electron diffraction patterns similar to LA and high concentrations of background dust interfere with fiber identification (NIOSH 7402, 1994). Lastly, the cost for TEM analysis is high relative to PLM, causing this study to cut down from 50 to 15 composite samples to be analyzed by TEM analysis.

9. Methods

9.1. Sampling Plan

The area sampled in Tubb Gulch consisted of 500 acres which was divided into 50, 10-acre plots. Originally 30 subsamples were collected in each 10-acre plot to create 1 composite sample as illustrated in Table I. However, due to the inclement weather and steep terrain, the 30 subsample requirement was reduced to 5 subsamples for each composite sample. All samples were grab samples and consisted of 500 g of material for each bark, duff, and soil. All the subsamples were collected co-located to one another. Soil samples were collected immediately under the duff sample location and if there was a tree close to the sampling area the bark sample was collected within the same area as the other two sub-samples. The samples analyzed by TEM were selected from positive PLM analysis samples. A global positioning system (GPS) was used to document the location of all the composite samples taken in the Tubb Gulch area

Table I: Sampling Plan

Sampling Plan			
Subsamples	Composite Samples	Composite Samples per 10-acre plot	
30	1	1	
Reduced			
5	1	1	
Total Bark Composite Samples (PLM-VE)	Total Duff Composite Samples (PLM-VE)	Total Soil Composite Samples (PLM-VE)	Total Composite Samples Each (TEM)
50	50	50	15
Total Samples Analyzed (PLM)	Total Samples Analyzed (TEM)	10-Acre Plots Analyzed (TEM)	
150	45	04, 11, 13, 17, 24, 26, 28, 31, 36, 40, 44, 46, 51, 52, 54	

The samples collected were selected to characterize the exposure units so the USFS could examine which areas contain the highest levels of LA. The eastern portion of the Tubb Gulch drainage falls within OU3 (Figure 5), the southern portion of the Tubb Gulch timber stands lie within OU2 (Figure 6), while the northernmost portion of the Tubb Gulch timber stands are outside of the USEPA designated boundaries for the NPL site (Figure 4) (Seccomb, 2015).

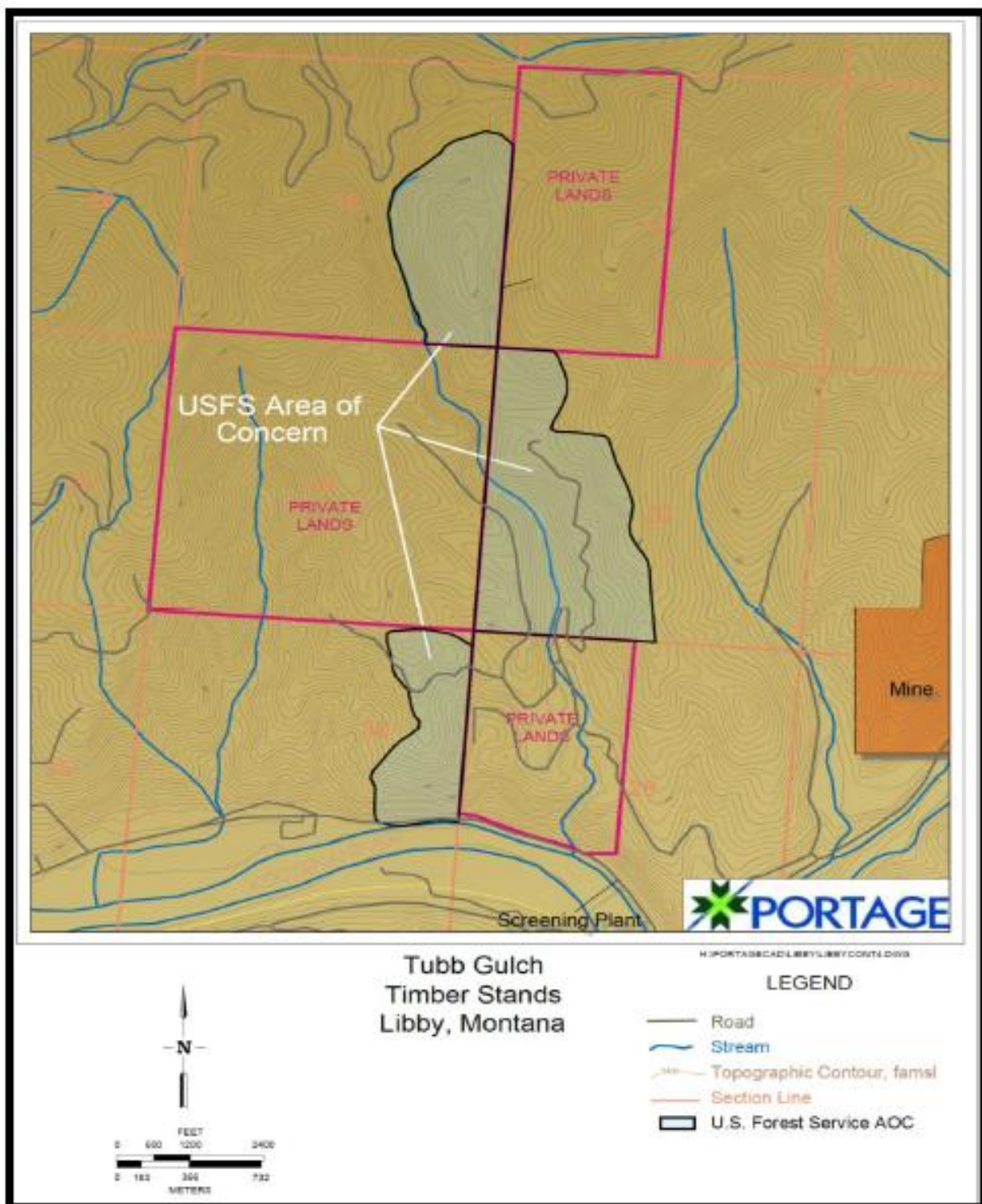


Figure 3: Tubb Gulch Sampling Areas

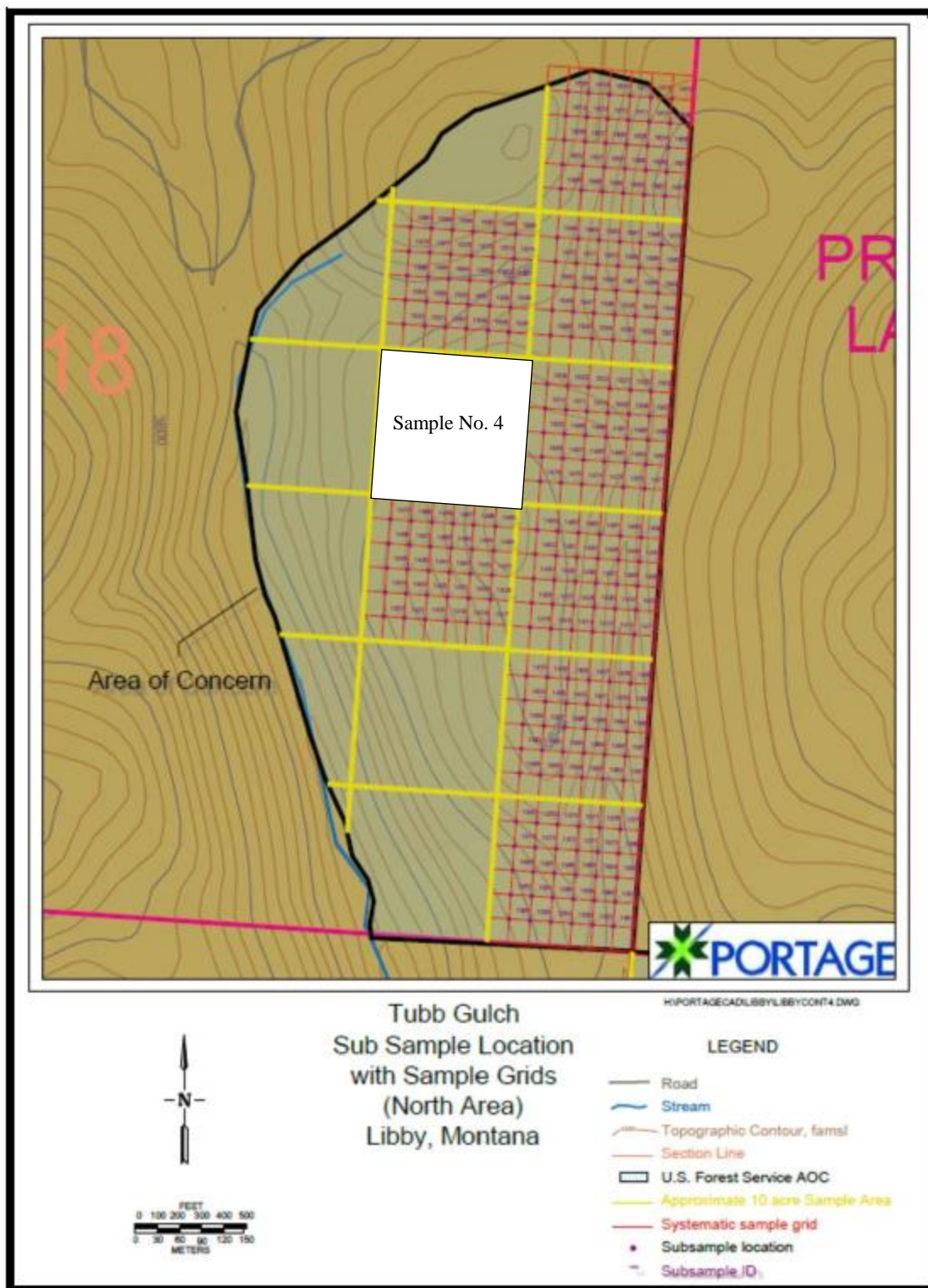


Figure 4: Tubb Gulch North Sampling Area

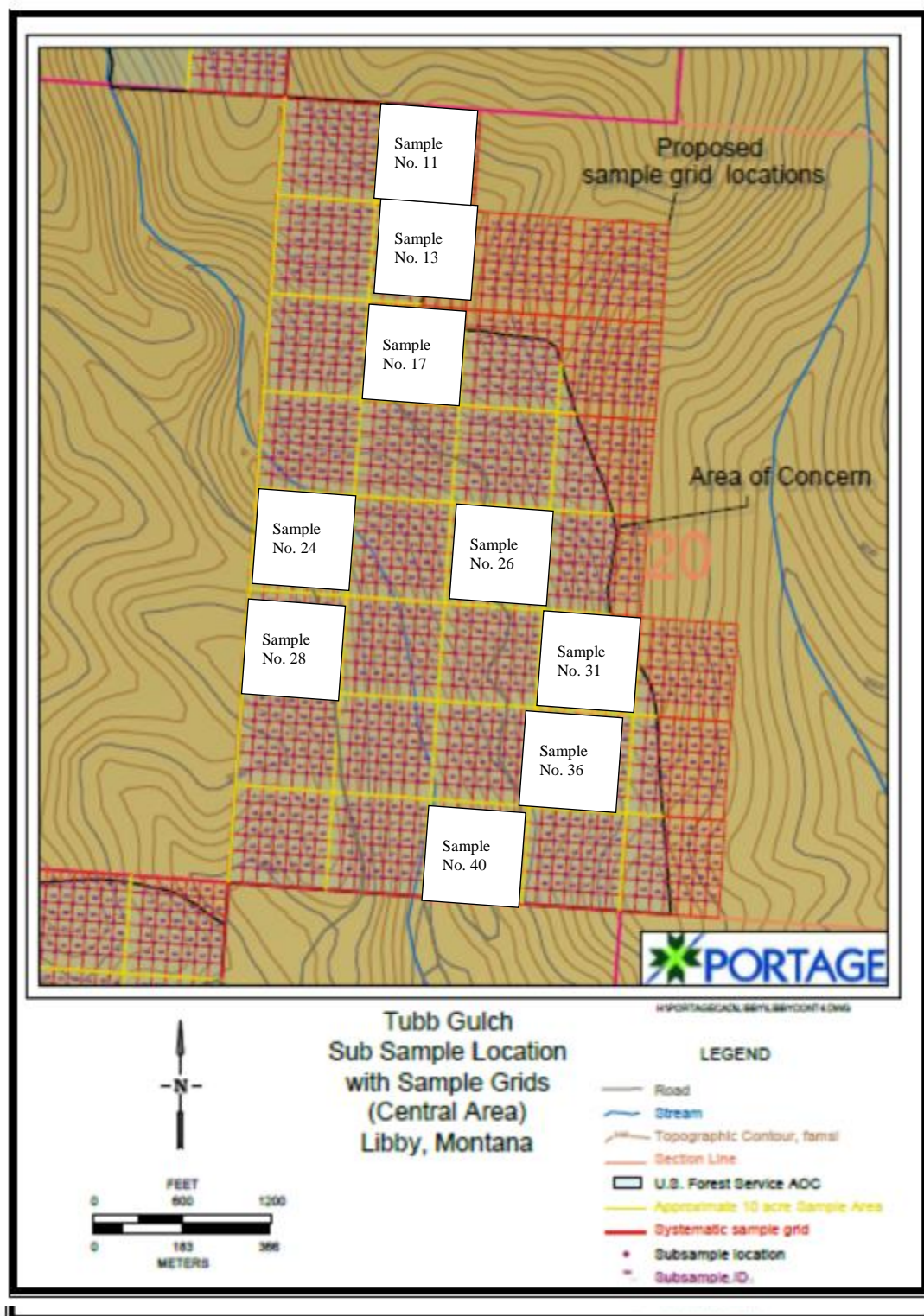


Figure 5: Tubb Gulch Central Sampling Area

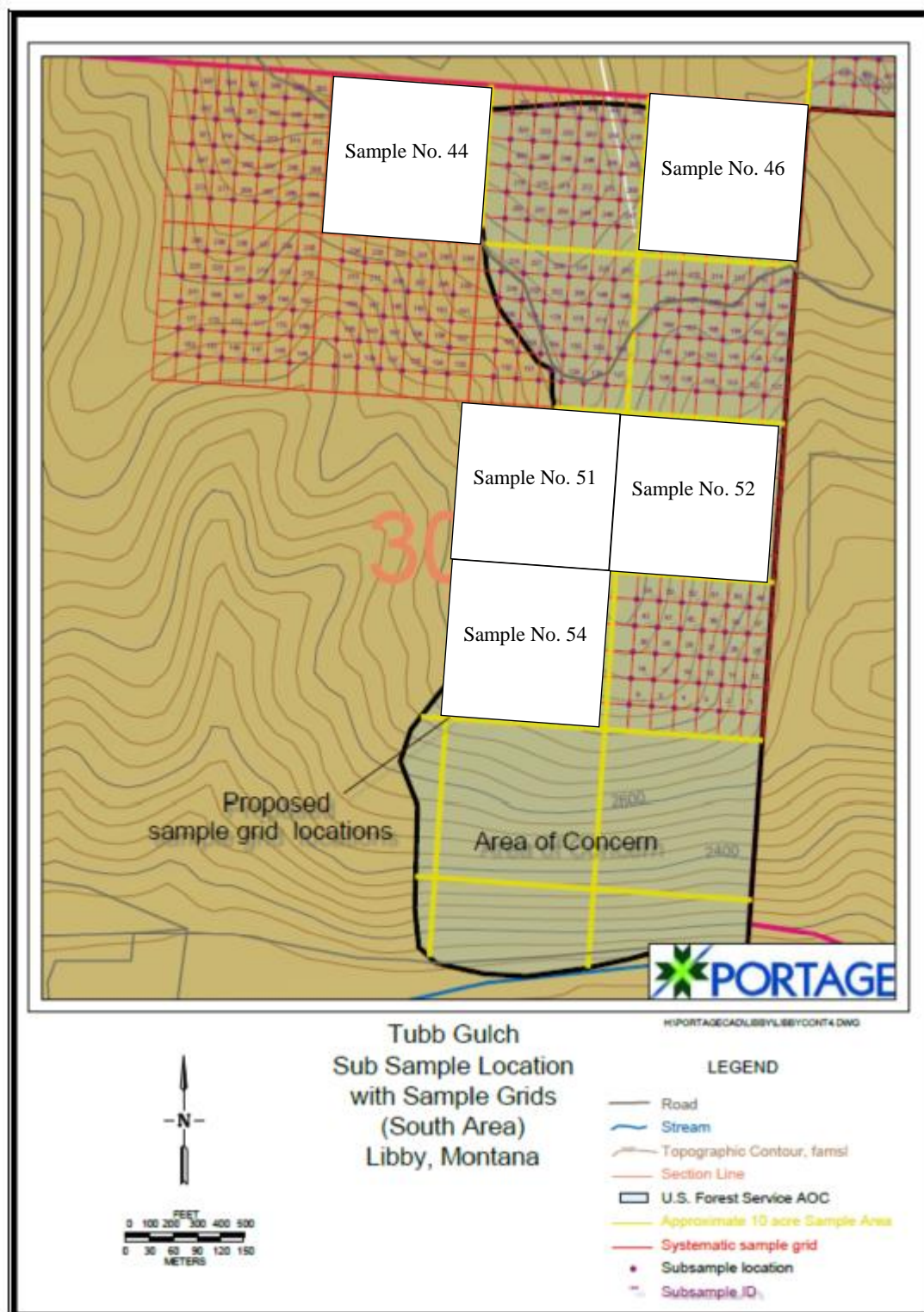


Figure 6: Tubb Gulch South Sampling Area

Preparation and initial screening of the composite samples for LA using PLM analysis was performed at the University of Montana – Ward Laboratory (UML) in Missoula, MT. The UML was chosen for this screening because of their experience with LA sampling and analysis.

The composite samples were then sent to EMSL Analytical Inc., in Cinnaminson, New Jersey to be analyzed by TEM method. The TEM analysis is used to determine representative concentrations of LA from the composite samples. Due to budgeting restraints, only 15 of the 50 bark, duff and soil composite samples analyzed by PLM were submitted for TEM analysis.

9.1.1. Bark Samples

Tree bark samples were collected from mature pines and firs at five locations in each 10-acre exposure unit co-located with duff and soil sampling. The selection of trees were based upon aspect, slope, and stand density which favored the deposition of LA. Samples were collected from trees which were a minimum of 15 inches in diameter. Bark samples were collected from the side of the tree facing toward the mine from a height of 4-5 ft. above ground level. These trees were present during the mining and milling operations, and are most likely to have been in contact with LA. The preferred tree species for sampling was Douglas Fir (*Pseudotsuga menziesli*). If Douglas Fir trees were not present in a timber stand because of elevation, aspect, or other conditions, another representative species of tree was chosen with preference given to Ponderosa Pine (*Pinus ponderosa*) or other rough bark species. Douglas firs were sampled because these tree species contained the roughest bark, which in turn would be a better reservoir for LA (Seccomb, 2015).

Each subsample was placed in a gallon-size zip seal plastic bag forming a composite sample consisting of 5 subsamples for the 10-acre exposure unit. A 2in drill bit and battery operated drill was used in extracting the bark samples from each tree (Seccomb, 2015).

9.1.2. Duff Samples

Duff samples, included any fresh or partially decayed organic debris (twigs, leaves, pine needles, etc.), were collected by hand from above the soil surface. The soil layer below the duff was not included in the duff sample. All duff and soil samples were collected co-located together. All subsamples were grab samples. Each subsample was placed in a gallon-size zip seal plastic bag and the mass of the resulting 5-subsample equaling one composite sample had to be no less than 500g (Seccomb, 2015).

9.1.3. Soil Samples

Co-located below the duff subsamples, the mineral soil layer was collected from 0-2in below the duff subsample. Each subsample was placed in a gallon sized zip seal plastic bag and the mass of the composite sample had to be no less than 500 grams (Seccomb, 2015).

9.1.4. Field Duplicate Samples

Field duplicate samples were collected as part of bark, duff, and soil sampling. A field duplicate is a field sample that is collected at the same place and time as an original field sample. Field duplicate pairs do not necessarily have the same or similar concentration values. Field duplicates help to evaluate variability due to small scale media heterogeneity, along with analytical precision (Seccomb, 2015). The field duplicates were not analyzed by TEM analysis.

9.2. PLM Analytical Method

The preparation and initial screening of composite samples for LA, using PLM analysis, was performed at UML. Pre-preparation for the samples began by heating the sample to 500 °C for 1 to 2 hours, or until all organic material was turned to ash. Then the PLM analysis was used to determine concentration of bulk samples. PLM analysis followed the guidelines of

EPA/600/R-93/116, OSHA method ID-191 (Crane, 1995), and NIOSH method 9002, which all involve similar procedures.

EPA Method 600/R-93/116 includes testing of bulk asbestos samples by performing a visual estimation (PLM-VE). This method is the most widely used for estimating asbestos in bulk samples. It requires more detailed method of point counting for accurate estimation of LA in samples with low concentrations. This method is not applicable for samples containing large amounts of fine fibers below the resolution of the PLM ($< 0.3 \mu\text{m}$) (EPA, 1993).

Polarized Gravimetric (PLM-Grav) analysis includes a detailed and very labor-intensive testing of bulk samples for organic components, inorganic acid-soluble and insoluble components. Analysis of sample can be performed by either PLM or TEM depending on the size of fibers expected in the sample (EPA, 1993b).

9.2.1. PLM Sample Pre-Preparation

NIOSH sampling method 9002 preparation began with pre-preparation which includes chemical reduction of the matrix, heating the sample to dryness or heating in the muffle furnace. The end result was a sample which has been reduced to a powder that was sufficiently fine to fit under a cover slip (NIOSH, 1994).

Samples with organic interference were placed in the muffle furnace. These samples included organic material that could be reduced by heating. The sample was removed from the bag and weighed in a balance to determine the weight of the submitted portion. The sample was then placed in the muffle at 500°C for 1 to 2 hours or until all obvious organic material had been removed. This was necessary to determine the asbestos content of the submitted sample (OSHA, 2016).

9.2.2. PLM Sample Preparation

If the sample had large lumps, was hard, or could not lie under a cover slip, the grain size had to be reduced. If the sample was powder or had been reduced enough, the sample would be ready to put on the slide glass. A glass slide was placed on a piece of optical tissue. Two drops of index of refraction medium were put on the slide. Powder from the sample was placed on the slide. A cover slip was placed over the medium and the sample (OSHA, 2016).

9.3. TEM Analytical Method

The TEM analysis quantified specific concentrations of LA in each of the composite samples and confirmed the range of LA concentrations in the Tubb Gulch area (Seccomb, 2015). When a sample is analyzed by TEM, the analyst records the size (length, width) with an aspect ratio $\geq 3:1$, have a length $\geq 0.5 \mu\text{m}$, and mineral type of each individual asbestos structure that is observed. Mineral type is determined by selected area electron diffraction (SAED) and energy dispersive spectroscopy (EDS), and each structure is assigned to one of the following four categories:

- LA Libby-class amphibole. Structures having an amphibole SAED pattern and an elemental composition similar to the range of fiber types observed in ores from the Libby mine (Meeker, 2003). This was a solid solution series of minerals including winchite and richterite, with lower amounts of tremolite.
- OA other amphibole-type asbestos fibers. Structures having an amphibole SAED pattern and an elemental composition that was not similar to fiber types from the Libby mine. Examples include crocidolite, amosite, and anthophyllite. There was no evidence that these fibers are associated with the Libby mine.

- CH Chrysotile fibers. Structured have a serpentine SAED pattern and an elemental composition characteristic of chrysotile. There was no evidence that these fibers were associated with Libby mine.
- NAM Non-asbestos material. These may include non-asbestos mineral fibers such as gypsum, glass, or clay, and may also include various types of organic and synthetic fibers derived from carpet, hair, etc.

(USEPA, 2014)

Transmission electron microscopy analysis was used to determine the concentration for samples containing LA. The USFS used TEM to confirm LA fibers in each positive sample from the PLM analysis. The TEM analysis was expensive and was used mainly as a conformation method in determining whether or not the PLM analysis could be used to detect traces of LA in the field.

10. Hypotheses Tests

Statistical analysis utilizing the hypothesis tests were used to test the validity of the claims made about the data. The software used to evaluate the hypothesis test was Minitab. Minitab provides a sample, effective way to input statistical data, manipulate that data, identify trends and patterns, and then extrapolate answers to the problem at hand (SixSigma, 2016). The claim that was trying to be verified is the null hypothesis (H_0). The alternative hypothesis (H_i) was accepted if the H_0 came back as untrue. The P-value from statistical hypothesis tests determine whether or not H_0 or H_i will be rejected. This value was a number between 0-1 and weights the strength of the evidence. A small P-value (≤ 0.05) indicates strong evidence against the H_0 , so H_0 is rejected. A large P-value (> 0.05) indicated weak evidence against the H_0 , so the H_0 was failed to be rejected. If the P-value was close to 0.05, the data was considered to be borderline and could go either way (Rumsey, 2011).

A statistical correlation test was used to determine if bark and duff had any areas related to one another from concentration. Three separate correlations were ran to compare concentrations for bark and duff (0.5-5 μm , $>5 \mu\text{m}$, and the total concentration). Statistical correlation results range from -1.0 to +1.0. This gives an indication of the strength of the relationship. The closer to ± 1.0 the stronger the relationship (Explorable, 2009).

11. Hypotheses

11.1. Bark

- H_0 : The majority of bark samples analyzed by PLM will not reveal asbestos structures by TEM analysis.
- H_i : The majority of bark samples analyzed by PLM will reveal asbestos structures by TEM analysis.

11.2. Duff

- H_0 : The majority of duff samples analyzed by PLM will not reveal asbestos structures by TEM analysis.
- H_i : The majority of duff samples analyzed by PLM will reveal asbestos structures by TEM analysis.

11.3. Relationship between Media Sources

- H_0 : There will not be a relationship between bark and duff sample LA concentrations measured at each sampling location.
- H_i : There will be a relationship between bark and duff sample LA concentrations measured at each sampling location.

12. Results and Discussion

A summary of the sampling results for bark, duff, and soil analyzed by PLM and TEM can be found in Tables II (Bark), Table III (Duff), and Table IV (Soil). Bark and duff were analyzed via PLM and TEM, and soil was analyzed via PLM-VE and PLM-Grav. All 50 samples for all three source media were analyzed by PLM-VE (Appendix A). However, due to budgetary constraints, only 15 of the 50 composite samples were analyzed by TEM for bark and duff only. The samples selected for TEM analysis were chosen from the positive PLM results. To be considered as positive PLM sample, the analysis for LA had to reveal concentrations above 1% (10,000 fibers/cc) (EPA, 2014b). Since positive PLM samples were selected for TEM analysis, only false positives may be identified. The results and discussion listed below will provide information on the hypothesis testing related to these results.

A qualitative description was conducted comparing PLM to TEM results for bark and duff separately. These data may be used to determine whether or not PLM could be used as a future primary analysis for bulk source media sampling. Secondly a correlation test was conducted to compare the TEM results between bark and duff to determine if there was an association between both. Lastly, a matrix scoring system was conducted for bark and duff concentrations, since they were recorded in separate units. The matrix scoring system was set up to compare concentration intensities.

Soil data was not graphically illustrated because TEM analysis was not conducted for soil samples. Therefore, the sample data was not expressed as concentrations but as traces (TR) or nondetect (ND) for LA.

12.1. Bark

Table II: Bark PLM and TEM Analysis Results

LA Concentrations in Tubb Gulch - Libby, Montana						
Bark						
Sample #	PLM-VE (University of Montana - Ward Lab)			TEM (EMSL Analytical Inc)		
	Non-Fibrous Components (%)	Non-Asbestos Fibrous Components (%)	Visual Estimate Asbestos Concentration	Fiber Length		Total Asbestos Concentration
				0.5 - 5.0 microns	> 5.0 microns	
	< 10,000 fibers/g	< 10,000 fibers/g		(Million structures/cm ²)	(Million structures/cm ²)	(Million structures/cm ²)
TG-BS-04	100	TR	TR	0.69	ND	0.69
TG-BS-11	100	0	TR	0.05	0.03	0.08
TG-BS-13	99	1	TR	0.12	0.08	0.20
TG-BS-17	100	TR	TR	0.35	0.11	0.46
TG-BS-24	100	TR	TR	0.76	0.46	1.22
TG-BS-26	100	TR	TR	0.21	ND	0.21
TG-BS-28	100	TR	TR	0.55	0.09	0.64
TG-BS-31	100	0	TR	0.43	0.13	0.56
TG-BS-36	100	2	TR	0.47	0.09	0.56
TG-BS-40	98	2	TR	0.34	ND	0.34
TG-BS-44	98	TR	TR	0.93	ND	0.93
TG-BS-46	100	TR	TR	1.39	0.50	1.89
TG-BS-51	100	TR	TR	ND	ND	ND
TG-BS-52	100	TR	TR	0.16	ND	0.16
TG-BS-54	100	TR	TR	0.96	0.18	1.14

Table II (bark) and Table III (duff) both have the same primary layout. These tables contain the results for PLM and TEM analysis. The first column is the sample number designated by the research team. The PLM analysis revealed three different sample results; the component of the sample consisting of non-fibrous materials, the component of the sample that revealed fibers, but not asbestos fibers (non-asbestos fibrous components) and the component of the sample that revealed an estimation of the asbestos concentration (visual estimation of asbestos concentration). These are illustrated as columns two, three and four, respectively. As discussed previously, a trace (TR) PLM concentration represents a sample revealing at least 1% or 10,000 fibers/cc of LA. If the concentration was below 1% the sample received a nondetect (ND).

Columns four through six pertain to TEM analyses. Column four illustrates the concentration of LA for fibers lengths of 0.5-5 microns and column five illustrates the concentration of LA for fiber lengths of 5 microns or greater. The final column six illustrates the total concentration (short and long fibers) of LA asbestos per sample.

It is important to note that the TEM concentrations for bark are expressed as fibers per surface area of bark (million structures/cm²) (Table II), while the TEM concentrations for duff are expressed as fibers per gram of duff (million structures/g) (Table III).

In terms of bark LA concentrations for fiber lengths of 0.5-5 microns, 14 of 15 PLM samples (93%) revealed positive TEM concentrations. For fiber lengths of 5 microns or greater, 9 of 15 PLM samples (60%) revealed positive TEM concentrations. These results suggest that the majority of samples analyzed by PLM revealed positive TEM LA concentrations. However, a fraction of PLM analyses may result in false positive samples when analyzed by TEM. Negative samples by PLM analysis would need to be analyzed by TEM to fully determine the precision of the PLM analysis.

Since the majority (93%) and (60%) of bark samples analyzed by PLM revealed positive TEM concentrations for asbestos structures 0.5-5 microns and asbestos structures ≥ 5 microns, we reject null hypothesis one.

12.2. Duff

Table III: Duff PLM and TEM Analysis Results

LA Concentrations in Tubb Gulch - Libby, Montana						
Duff						
Sample #	PLM-VE (University of Montana - Ward Lab)			TEM (EMSL Analytical Inc)		
	Non-Fibrous Components (%)	Non-Asbestos Fibrous Components (%)	Visual Estimate Asbestos Concentration	Fiber Length		Total Asbestos Concentration
				0.5 - 5.0 microns	> 5.0 microns	
	< 10,000 fibers/g	< 10,000 fibers/g		(Million structures/gram)	(Million structures/gram)	(Million structures/gram)
TG-DS-04	85	15	TR	40	5	45
TG-DS-11	75	25	TR	119	22	141
TG-DS-13	85	15	TR	127	19	146
TG-DS-17	85	15	TR	51	22	73
TG-DS-24	85	15	TR	34	3	37
TG-DS-26	80	20	TR	44	30	74
TG-DS-28	85	15	TR	173	22	195
TG-DS-31	85	15	TR	256	59	315
TG-DS-36	88	12	TR	182	7	189
TG-DS-40	85	15	TR	235	38	273
TG-DS-44	88	12	TR	13	ND	13
TG-DS-46	85	15	TR	133	12	145
TG-DS-51	85	15	TR	134	30	164
TG-DS-52	85	15	TR	175	24	199
TG-DS-54	85	15	TR	169	29	198

Duff sample results are presented in Table III. For fiber lengths of 0.5-5 microns, 15 of 15 PLM samples (100%) revealed positive TEM concentrations. For fiber lengths of 5 microns or greater, 14 of 15 samples (93%) revealed positive TEM concentrations. These results suggest that a large majority of duff samples analyzed by PLM revealed positive TEM LA concentrations. Since the majority (100%) and (93%) of duff samples analyzed by PLM revealed positive TEM concentrations for asbestos structures 0.5-5 microns and asbestos structures ≥ 5 microns, we reject null hypothesis two.

Initially, a Pearson's correlation analysis was considered to determine if a correlation existed between TEM analytical results for bark and duff data. It was anticipated that since the

samples were co-located, there may be a correlation observed. However, since the TEM concentrations for bark were expressed as fibers per surface area of bark (million structures/cm²) and the TEM concentrations for duff were expressed as fibers per gram of duff (million structures/g) this statistical test could not be used.

Tree bark and duff TEM analysis results for short and long asbestos structures (0.5-5 μ m and >5 μ m) were graphed and are presented in Figures 7 and 8 below. The smaller fibers (0.5-5 μ m) were reported in higher concentrations in both media sources. This is consistent with previous studies (Hart et al., 2007; Hart et al., 2009; Ward et al., 2009). It is hypothesized that shorter fibers were more likely to be suspended by the wind.

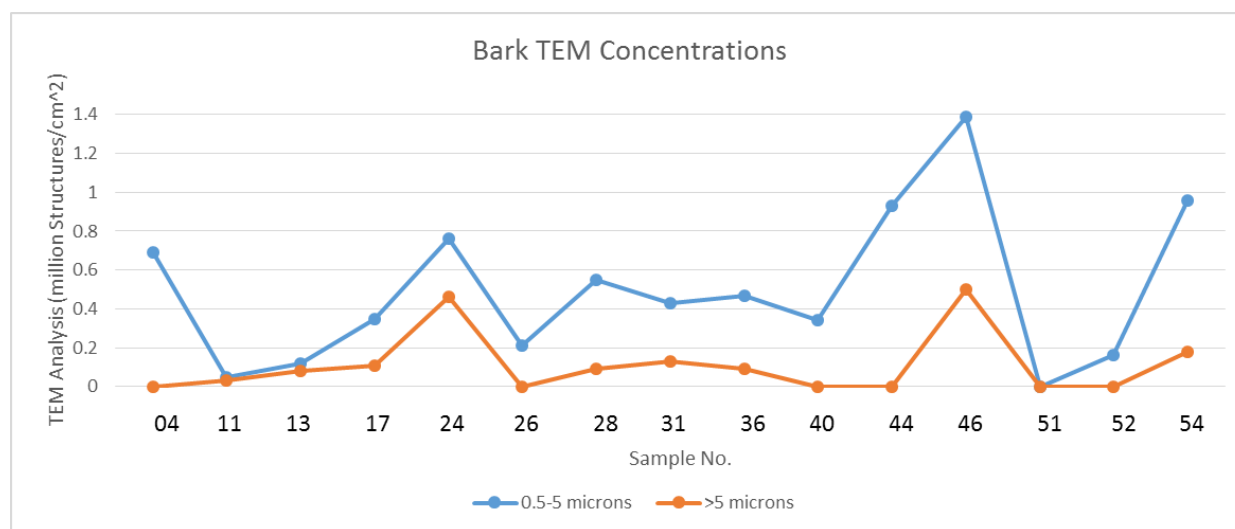


Figure 7: Bark Samples TEM Analysis Concentration Results

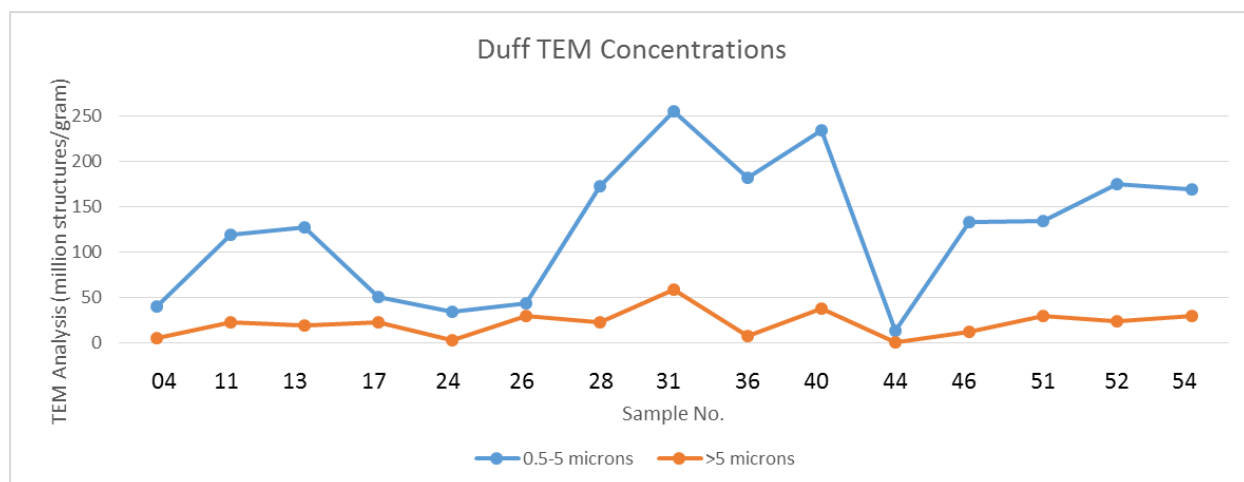


Figure 8: Duff Samples TEM Analysis Concentration Results

As noted in the previous section, a major limitation in performing a correlation assessment for bark and duff was that bark concentrations were expressed as million structures/cm² and duff concentrations were expressed as million structures/gram. Since bark and duff were presented in separate units, a matrix scoring system (Table IV) was developed to compare the concentrations as a qualitative comparison instead of quantitative, for both source media. Only the total concentration for bark and duff were used for the matrix scoring system (Table V). The matrix ranges from 10-0. A value of zero was assigned to samples revealing a non-detect (ND) TEM concentration. Bark was scored by increments of 0.2 million structures/cm² and duff was scored by increments of 32 million structures/gram. The total concentrations for each sample was given a number that closely represented the increments. For example, bark sample TG-BS-04 total concentration was 0.69 so that received a score of 3 and for duff sample TG-DS-04 total concentration was 45 so that received a score of 2 and so on. This system allowed both sample media to be compared. The trend of the samples (Figure 9) may be used as a visual estimate of a correlation, showing on average when bark concentrations were high then duff concentrations were low and vice versa.

Table IV: Matrix Scoring System for Bark and Duff Concentrations

Matrix Scoring System for Concentrations		
Score System	Bark	Duff
0	ND	ND
1	0.01-0.29	1.0-32
2	0.3-0.49	33-64
3	0.5-0.69	65-96
4	0.7-0.89	97-128
5	0.9-1.09	129-160
6	1.1-1.29	161-192
7	1.3-1.49	193-224
8	1.5-1.69	225-256
9	1.7-1.89	257-288
10	1.9-2.0	289-320

Table V: Matrix Scores for Bark and Duff Concentration Results

Matrix Scores for Concentration Results				
Sample #	Bark Total Concentration Results	Bark Matrix Score	Duff Total Concentration Results	Duff Matrix Score
4	0.69	3	45	2
11	0.08	1	141	5
13	0.2	1	146	5
17	0.46	2	73	3
24	1.22	6	37	2
26	0.21	1	74	3
28	0.64	3	195	7
31	0.56	3	315	10
36	0.56	3	189	6
40	0.34	2	273	9
44	0.93	5	13	1
46	1.89	9	145	5
51	ND	0	164	6
52	0.16	1	199	7
54	1.14	6	198	7

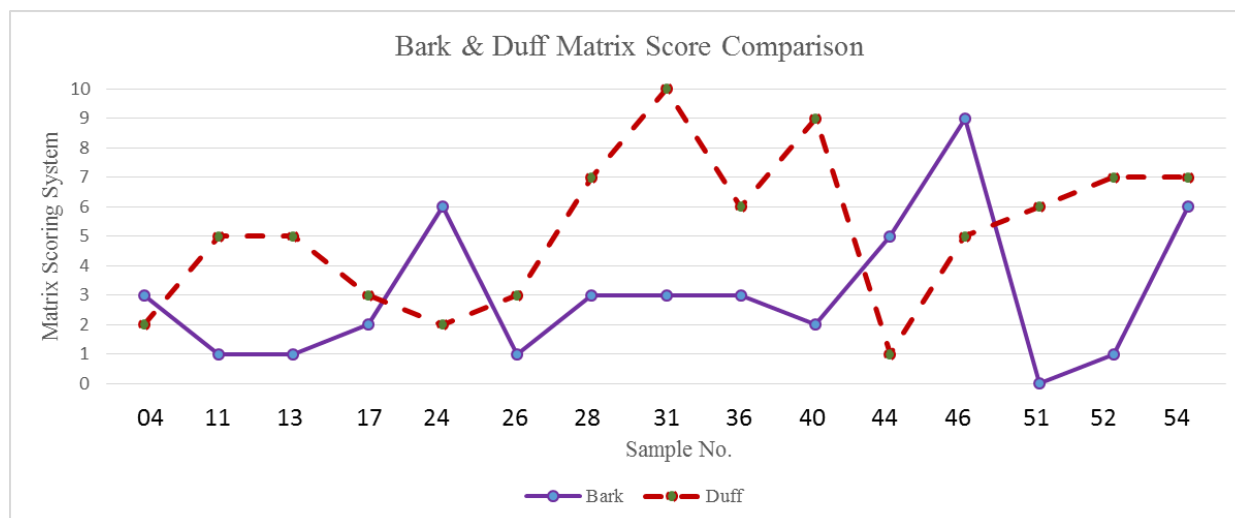


Figure 9: Matrix Scoring System for Bark and Duff Comparison

As illustrated in Figure 9, there does not appear to be a relationship between LA concentrations for bark and duff, even though samples were collected in the same location. The matrix ranking with regards to the sample locations are shown on Figures 10 (bark) and 11 (duff). Since no relationship was observed between bark and duff sample LA concentrations measured at each sampling location, we fail to reject null hypothesis 3.

The matrix ranking system was broken down further into four distinct sample ranges: red (10-7), green (6-4), brown (3-1), and white (0). Red was used for the higher concentrated areas and white was used for the ND areas. In figures 10 and 11, it should be noted that the mine site sits slightly south-east from the middle sampling areas. The concentrations illustrated in these figures show that the distance from the mine did not necessarily predict the concentrations of LA contamination.

In Figure 10 bark results show low rankings for composite samples closest to the mine and also a ND next to a red ranking composite sample. Rankings did not correlate to distance away from the mine and more TEM analysis would need to be assessed to fully characterize the

area. For Figure 11 duff results followed a typical trend where the higher ranking results were located closer to the mine. A TEM analysis would need to be ran on all 50 samples for each category (bark, duff, and soil) to fully characterize the area.

Possible reasoning to explain the characteristics of the samples would be prevailing wind direction. Referring back to Figure 1, the prevailing wind direction was north to north-east causing the contamination for the mine to be blown away from the area sampled in Tubb Gulch. Dependent on the wind direction the area sampled (Figure 10 and 11) would most likely be contaminated from transporting the vermiculite on the highway (OU8) or even from the expansion facility (OU1) located in the town of Libby. More sampling is recommended to validate this hypothesis.

Another hypothesis could be that may explain this outcome was that Douglas Fir trees had shed their bark causing fibers embedded in the bark to become duff. The majority of trees sampled were Douglas Fir. If an appropriate Douglas Fir tree was not in the sampling area, a Ponderosa Pine tree was chosen for sampling. That idea would make sense when bark concentrations were low and duff high. However, according to Pelt, 2005, Douglas fir trees are the most fire-resistant of all the trees native to north western portion of the US, due to the protective bark that develops as the tree ages. Douglas fir produce large amounts of bark which it retains for a long period of time (Pelt, 2005). This shows that bark should be relatively close to duff concentrations if this hypothesis was true.

A third possible hypothesis could be that samples were taken in May during the rainy season. This could have caused bark to contain lower LA concentrations, due to washout, but not all bark samples came back with low concentrations. Then during the dry seasons, the bark concentrations could increase due to wind disturbing the duff and soil area, dispersing the LA

fibers back into the air and would impact on the tree bark. Seasonal sampling would have to be conducted to confirm this hypothesis.

Tubb Gulch - Libby Montana
PLM Bark Screening Results
7/23/2015

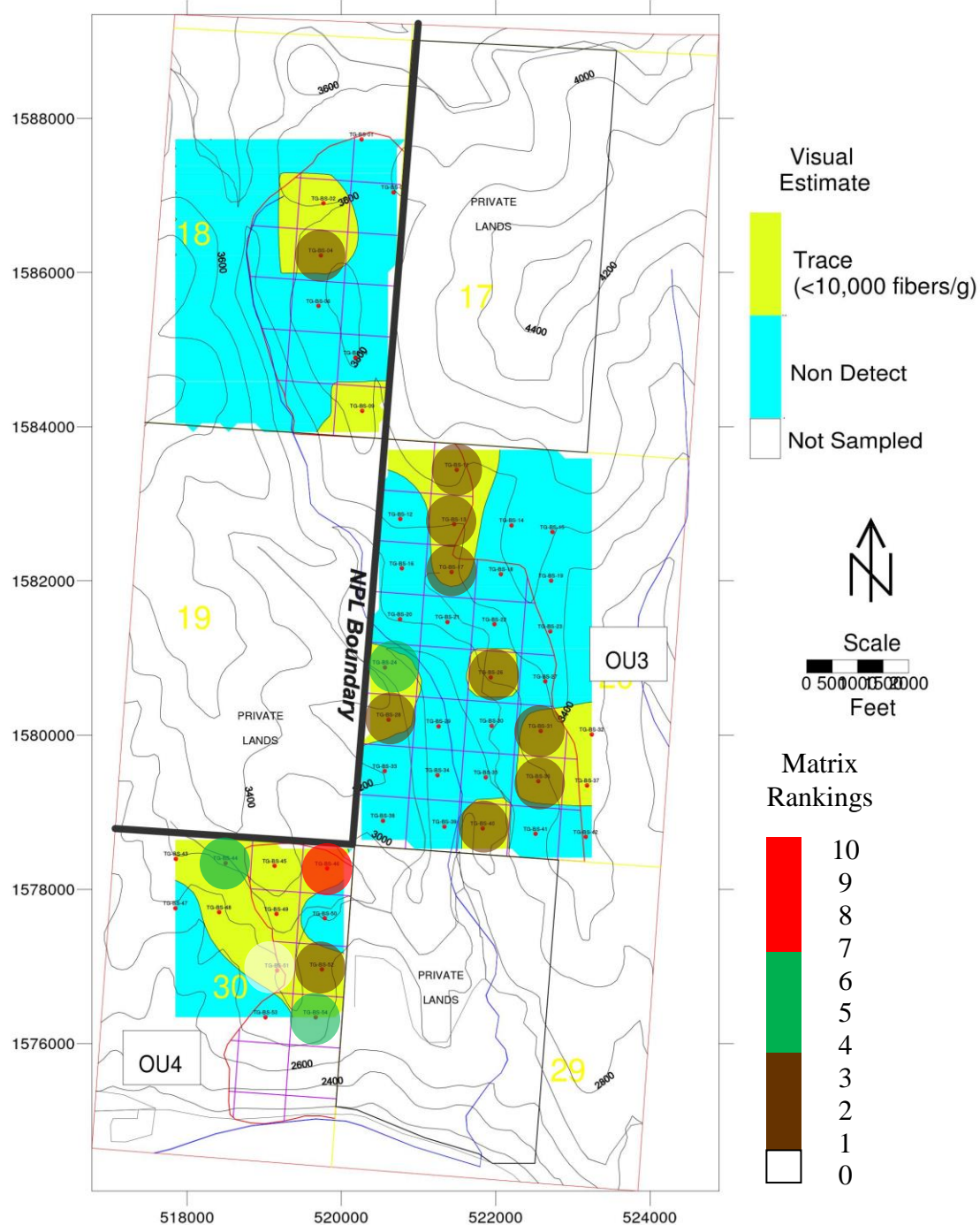
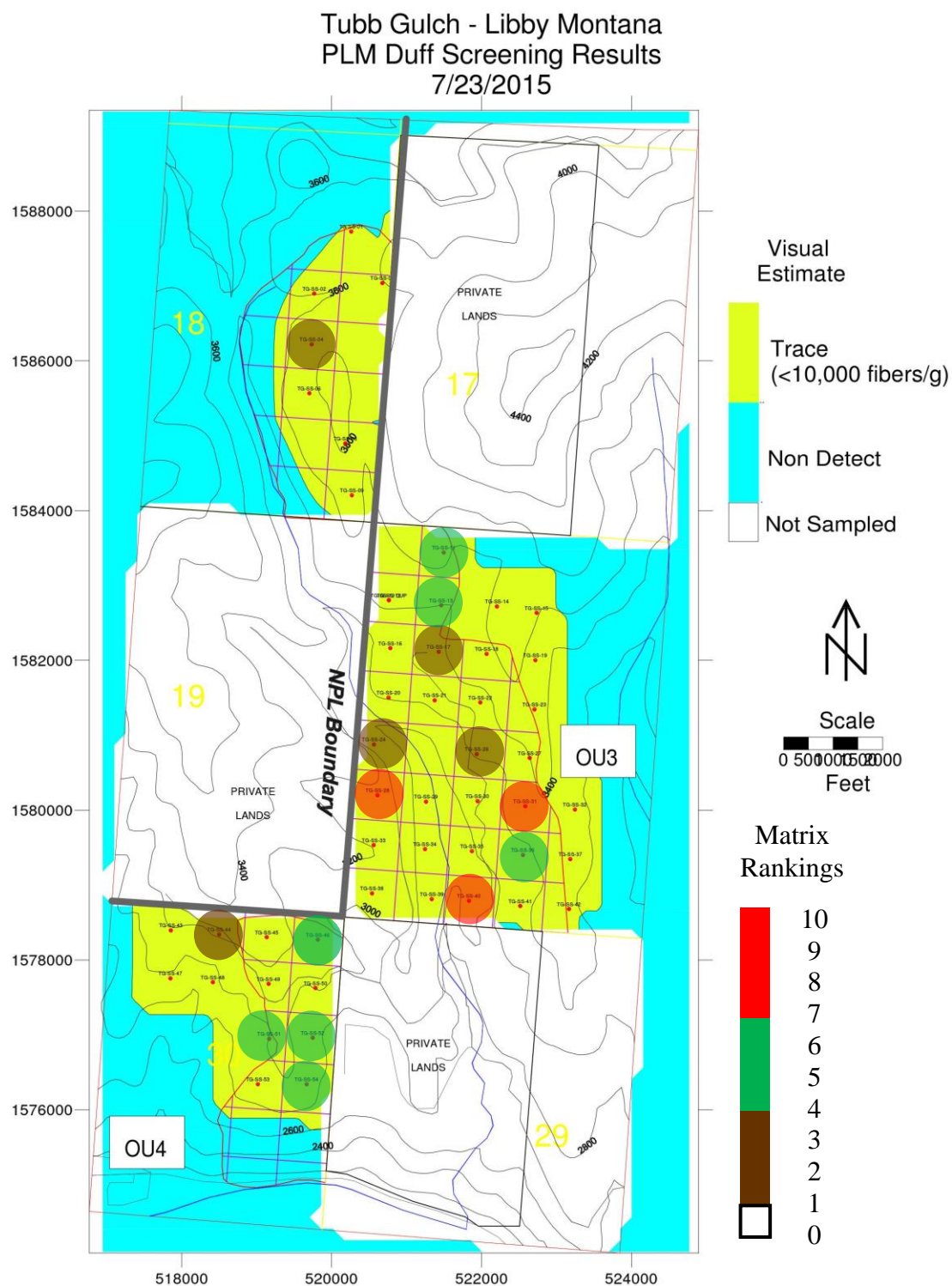


Figure 10: Bark Topography Matrix Rankings



12.3. Soil

Table VI: Soil PLM Analysis Results

LA Concentrations in Tubb Gulch - Libby, Montana					
Soil					
Sample No.	PLM-VE (University of Montana - Ward Lab)			Semi-Quantitative PLM results (EMSL Analytical Inc)	
	Non-Fibrous Components (%)	Non-Asbestos Fibrous Components (%)	Visual Estimate Asbestos Concentration	PLM-Grav	PLM-VE
	< 10,000 fibers/g	< 10,000 fibers/g		Fine Fraction (< 1/4 inch)	Coarse Fraction (> 1/4 inch)
TG-SS-04	85	15	TR	ND	ND
TG-SS-11	75	25	TR	ND	ND
TG-SS-13	80	20	TR	ND	ND
TG-SS-17	80	20	TR	ND	ND
TG-SS-24	85	15	TR	ND	ND
TG-SS-26	80	20	TR	ND	ND
TG-SS-28	88	12	TR	ND	ND
TG-SS-31	85	15	TR	ND	ND
TG-SS-36	75	25	TR	ND	ND
TG-SS-40	80	20	TR	ND	ND
TG-SS-44	85	15	TR	ND	ND
TG-SS-46	85	15	TR	ND	ND
TG-SS-51	90	10	TR	ND	ND
TG-SS-52	88	12	TR	ND	ND
TG-SS-54	88	12	ND	ND	ND

Soil composite sample results are presented in Table VI. Since soil samples were analyzed by two separate PLM analyses, and not TEM, these data were not considered for hypothesis testing. It is interesting to note that the soil analytical methods, UML PLM-VE, EMSL PLM-VE or PLM-Grav revealed substantially different results. A TEM analysis would need to be used to justify positive samples for LA fibers. However, due to budgetary constraints soil composite samples were not analyzed by TEM.

13. Conclusion

The research for this thesis was focused around evaluating (LA) concentrations from bark and duff composite samples. The composite samples for bark and duff were evaluated first by PLM analysis and 15 positive PLM samples were further analyzed by TEM. The majority (100%) and (93%) of duff samples analyzed by PLM revealed positive TEM concentrations for asbestos structures 0.5-5 microns and asbestos structures ≥ 5 microns and, the majority (93%) and (60%) of bark samples analyzed by PLM revealed positive TEM concentrations for asbestos structures 0.5-5 microns and asbestos structures ≥ 5 microns. These data suggest that PLM analysis may be a reliable LA screening method for bark and duff samples. However, it is important to note that only positive PLM samples were submitted for TEM analysis; therefore, the potential for false negative PLM sample results was not assessed with this research.

Since the PLM method of identification was limited to fibers approximately 1 μm in diameter or thicker (Dodson & Hammar, 2011), PLM analysis becomes less accurate in being a primary LA detection method, but could not be tested since only positive PLM analysis composite samples were selected for analysis. The TEM analysis results suggest that PLM analysis can obtain false positives mainly in the larger LA fiber sizes ($>5.0\mu\text{m}$). The highest concentration of LA fibers detected from TEM analysis were in the category of 0.5-5 μm . The quantification of false negatives could have been analyzed, but because of budgetary constraints only 15 positive PLM composite samples for bark and duff were sent to the lab for TEM analysis for validation.

The results of this study were valuable in further characterizing the Tubb Gulch area. All bark and duff composite samples revealed the presence of asbestos structures via TEM with the exception of one bark sample. Based on these source media results, there is a potential for LA

exposure to USFS personnel or members of the public when working or recreating in the Tubb Gulch area. The full extent of the LA contamination is unknown and additional sampling is recommended to fully understand the total impact of contamination, not only in the Tubb Gulch area but in areas further from the mine and town of Libby.

To minimize exposure to LA, level C personal protective equipment (PPE) is recommended for those performing work in and around the Tubb Gulch area. Level C PPE consists of full body Tyvek, double gloves, boots and a full faced positive pressure respirator all taped to prevent an opening for LA to enter. To better understand the full extent of the inhalation hazard to USFS personnel or the public, activity based sampling will need to be performed.

References Cited

- ATSDR. (2003). Public health assessment: Libby asbestos NPL site OU4: Screening plant, export plant, town of libby, and affected libby valley residential and commercial properties lincoln county, montana EPA facility ID: MT0009083840 (Public Health Assessment. Atlanta, GA: U.S. Department of Health and Human Services Agency for Toxic Substances and Disease Registry.
- ATSDR. (2008). Exposure to asbestos-containing vermiculite from libby, montana, at 28 processing sites in the united states (Summary Report
- ATSDR. (2010). Mortality from asbestosis in libby, montana, 1979-1998 Agency for Toxic Substances and Disease Registry.
- Case, B.W., Abraham, J.L., Meeker, G., Pooley, F.D., and Pinkerton, K.E. (2011). Applying definitions of “Asbestos” to environmental and “Low-dose” exposure levels and health effects, particularly malignant mesothelioma.
- Crane, T. D. (1995). Polarized light microscopy of asbestos (Technical Report. Salt Lake City, Utah: Branch of Physical Measurements and Analysis.
- Dodson, F. Ronald, Hammar, P. Samuel. (2011). In Editors Ronald Dodson and Samuel Hammar (Ed.), Asbestos: Risk assessment, epidemiology, and health effects (Second ed.). Boca Raton, FL: CRC Press.
- EPA. (1993b). Method for the determination of asbestos in bulk building materials (Technical Report US Department of Commerce National Technical Information Service.

EPA. (1994). Methods for derivation of inhalation reference concentrations and application of inhalation dosimetry No. 600/8-90/066F). Washington DC: Office of Research and Development.

EPA. (2007). LIBBY ASBESTOS SUPERFUND SITE, OPERABLE UNIT 3 DATA SUMMARY REPORT: 2007 TO 2013 revision 2 – january 2015 (Data Summary. Denver, CO: CDM Smith.

EPA. (2013). Baseline ecological risk assessment for non-asbestos contaminants operable unit 3 libby asbestos superfund site libby, montana CDM Federal Programs Corporation.

EPA. (2013b). Summary of published measurements of asbestos levels in ambient air. Denver, CO: SRC Inc.

EPA. (2013c). Sampling and analysis plan/ quality assurance project plan operable unit 3, libby asbestos superfund site. Denver, CO: U.S. Environmental Protection Agency.

EPA. (2014). Site-wide human health risk assessment (Executive Summary CDM Federal Programs Corporation.

EPA. (2014b). Toxicological review of libby amphibole asbestos. Washington, DC: U.S. Environmental Protection Agency.

EPA. (2016). EPA announces final cleanup plan for libby and troy (Record of Decision Montana Department of Environmental Quality.

Explorable. (2009). Statistical correlation. Retrieved May 17, 2016,

from <https://explorable.com/statistical-correlation>

Hart, J.F., Ward, T.J., Spear, T.M., Crispen, K., and Zolnikov, T.R., 2007. Evaluation of asbestos exposures during firewood harvesting simulations in Libby, Montana, *Annals of Occupational Hygiene*, 51, 8: 1-7.

Hart, J.F., Spear, T.M., Ward, T.J., Baldwin, C. E., Salo, M.N., and Elashheb, M.I., 2009. An evaluation of the potential exposure to asbestiform amphiboles near a former vermiculite mine. *Journal of Environmental and Public Health*, Article ID 189509, 10 pages.
doi:10.1155/2009/189509.

Hestmark, M. (2015). Five- year review report: Libby montana superfund site (Review Report. Denver, CO: US Environmental Protection Agency.

Hinds, W. C. (1999). 20.5 asbestos counting. *Aerosol technology: Properties, behavior, and measurement of airborne particle* (2nd ed.,). Canada: John Wileyand Sons.

IRIS. (2014). Libby amphibole asbestos; CASRN not applicable (Chemical Assessment Summary Report

Lemieux, P. (2012). Emissions of amphibole asbestos from the simulated open burning of duff from libby, MT. Research Triangle Park, NC: EPA.

Meeker, G. P., Bern, A. M., Brownfield, I. K., Lowers, H. A., Sutley, S. J., Hoefen, T. M., et al. (2003). The composition and morphology of amphiboles from the rainy creek complex, near

libby, montana

American Mineralogist, 88(11-12 PART 2), 1955.

NIOSH. (2015). Appendix C - supplementary exposure limits (Chemical Hazards Exposure Limits. Atlanta, GA: CDC.

NIOSH 7402. (1994). Asbestos by TEM (Manual of Analytical Methods No. 4th)CDC.

NIOSH 9002. (1994). ASBESTOS (bulk) by PLM No. Fourth Edition)NIOSH Manual of Analytical Methods (NMAM).

OSHA. (2016). Polarized light microscopy of asbestos - non-mandatory No. 1915.1001 App K)

OSHA 1910.1001. (2013). Polarized light microscopy of Asbestos 1910.1001 app J (Occupational Safety and Health Standards

Pelt, R. V. P. (2005). Identifying mature and old forests in western washington: Douglas fir (*pseudotsuga menziesii*). Washington State Department of Natural Resources,

Perkins, R. L., & Harvey, B. W. (1993). Method for the determination of asbestos in bulk building materials (Test Method No. 68024550). Research Triangle Park, NC: U.S. EPA.

Rumsey, D. J. (2011). What a p-value tells you about statistical data (978-0-470-91108-2 ed.) John Wiley and Sons inc.

Seccomb, P. (2015). Tubb gulch site investigation work plan lincoln county, montana (Sampling and Analysis Work Plan No. AG-02RC-C-14-0047)

SixSigma. (2016). What is minitab? Retrieved 7/15, 2016, from <http://www.6sigma.us/what-is-minitab/>

Sullivan, P. A. (2007). Vermiculite, respiratory disease, and asbestos exposure in libby, montana: Update of a cohort mortality studyEnvironmental Health Prospect.

Ward T, Spear T, Hart J, Noonan C, Holian A, Getman M, Webber J. 2006. Trees as reservoirs for amphibole fibers in Libby, Montana, Science of the Total Environment; 367(Aug. 15):460-465.

Ward, T.J., Hart, J.F., Spear, T.M., Meyer, B.J., and Webber, J.S., 2009. Fate of Libby amphibole fibers when burning contaminated firewood, Environmental Science & Technology, 43(8) 2878–2883. PMCID: PMC2688714.

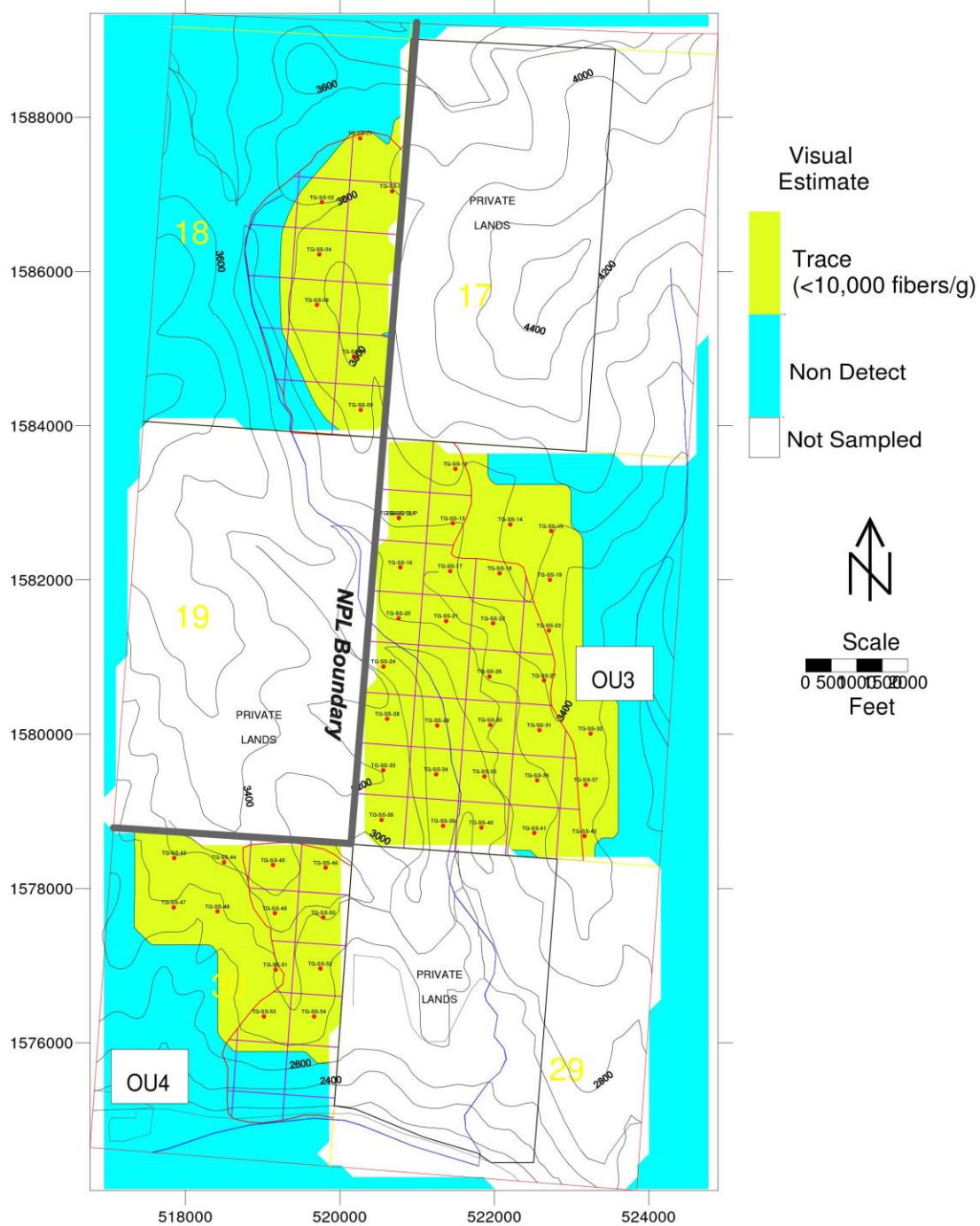
Ward, T.J., Spear, T.M., Hart, J.F., Webber, J.S., and Elashheb, M.I., 2012. Amphibole asbestos in tree bark – a review of findings for this inhalational exposure source in Libby, Montana. Journal of Occupational & Environmental Hygiene, 9: 387–397.

Appendix A:

PLM Results: Duff

Portage Samples - Duff									
Reservoirs Environmental Inc Results									
August 12, 2015									
						Reservoirs Results			
Sample ID	Portage Collection Date	Portage Collection Time	Media Type	Ash Date	Final Mass of Ash (g)	Mineral	Visual Estimate (%)	Non Asbestos Fibrous Components (%)	Non-Fibrous Components (%)
TG-DS-01	5/14/2015	900	Duff	6/14/2015	38.1104	Trem/Act	TR	20	80
TG-DS-01 DUP	5/14/2015	900	Duff	6/14/2015	27.508	Trem/Act	TR	15	85
TG-DS-02	5/15/2015	1000	Duff	6/8/2015	47.3335	Trem/Act	TR	15	85
TG-DS-03	5/14/2015	1000	Duff	6/13/2015	31.764	Trem/Act	TR	15	85
TG-DS-04	5/15/2015	1100	Duff	6/7/2015	24.0993	Trem/Act	TR	15	85
TG-DS-06	5/15/2015	1200	Duff	6/8/2015	34.111	Trem/Act	TR	25	75
TG-DS-08	5/15/2015	1100	Duff	6/11/2015	11.472	Trem/Act	TR	15	85
TG-DS-09	5/15/2015	1000	Duff	6/10/2015	19.687	Trem/Act	TR	12	88
TG-DS-11	5/19/2015	0800	Duff	5/30/2015	27.278	Trem/Act	TR	25	75
TG-DS-12	5/20/2015	0730	Duff	6/2/2015	20.4656	Trem/Act	TR	15	85
TG-DS-12 DUP	5/20/2015	0730	Duff	6/6/2015	25.5886	Trem/Act	TR	15	85
TG-DS-13	5/19/2015	0730	Duff	6/1/2015	22.3081	Trem/Act	TR	15	85
TG-DS-14	5/19/2015	0830	Duff	5/31/2015	42.1447	Trem/Act	TR	20	80
TG-DS-15	5/19/2015	0900	Duff	5/30/2015	18.1099	Trem/Act	TR	15	85
TG-DS-16	5/20/2015	0800	Duff	6/6/2015	25.1577	Trem/Act	TR	20	80
TG-DS-17	5/19/2015	1000	Duff	5/29/2015	24.2334	Trem/Act	TR	15	85
TG-DS-18	5/19/2015	1100	Duff	5/30/2015	30.6373	Trem/Act	TR	15	85
TG-DS-19	5/19/2015	0930	Duff	5/29/2015	53.2498	Trem/Act	TR	15	85
TG-DS-20	5/20/2015	0830	Duff	6/6/2015	98.2335	Trem/Act	TR	8	92
TG-DS-21	5/19/2015	1100	Duff	6/2/2015	35.1317	Trem/Act	TR	20	80
TG-DS-22	5/19/2015	1030	Duff	6/1/2015	37.1652	Trem/Act	TR	15	85
TG-DS-23	5/19/2015	1000	Duff	5/31/2015	56.6058	Trem/Act	TR	25	75
TG-DS-24	5/20/2015	0800	Duff	6/5/2015	15.4024	Trem/Act	TR	15	85
TG-DS-26	5/18/2015	1130	Duff	6/4/2015	25.5895	Trem/Act/Chrysotile	TR	20	80
TG-DS-27	5/18/2015	1100	Duff	6/12/2015	45.6091	Trem/Act	TR	20	80
TG-DS-28	5/20/2015	0830	Duff	6/7/2015	11.7417	Trem/Act	TR	15	85
TG-DS-29	5/18/2015	0830	Duff	6/14/2015	21.719	Trem/Act	TR	15	85
TG-DS-30	5/18/2015	0800	Duff	6/3/2015	24.2093	Trem/Act	TR	15	85
TG-DS-31	5/18/2015	1030	Duff	6/13/2015	32.7644	Trem/Act	TR	15	85
TG-DS-32	5/18/2015	1000	Duff	6/12/2015	38.8614	Trem/Act	TR	15	85
TG-DS-33	5/20/2015	0900	Duff	6/1/2015	23.3204	Trem/Act	TR	15	85
TG-DS-34	5/18/2015	0930	Duff	6/11/2015	57.6436	Trem/Act	TR	15	85
TG-DS-35	5/19/2015	0800	Duff	5/31/2015	19.3549	Trem/Act	TR	15	85
TG-DS-36	5/18/2015	0800	Duff	6/3/2015	58.9712	Trem/Act	TR	12	88
TG-DS-37	5/18/2015	0930	Duff	6/4/2015	20.4308	Trem/Act	TR	15	85
TG-DS-38	5/20/2015	0930	Duff	6/5/2015	19.1603	Trem/Act	TR	15	85
TG-DS-39	5/18/2015	1030	Duff	6/13/2015	39.2254	Trem/Act	TR	15	85
TG-DS-40	5/19/2015	0900	Duff	6/2/2015	35.9067	Trem/Act	TR	15	85
TG-DS-41	5/18/2015	0830	Duff	6/8/2015	42.539	Trem/Act	TR	20	80
TG-DS-42	5/18/2015	0900	Duff	6/4/2015	40.028	Trem/Act	TR	15	85
TG-DS-43	5/16/2015	1000	Duff	5/28/2015	28.415	Trem/Act	TR	10	90
TG-DS-44	5/16/2015	1030	Duff	6/10/2015	79.6787	Trem/Act	TR	12	88
TG-DS-45	5/16/2015	1100	Duff	6/9/2015	72.8878	Trem/Act	TR	15	85
TG-DS-46	5/16/2015	1130	Duff	6/9/2015	10.513	Trem/Act	TR	15	85
TG-DS-47	5/17/2015	1030	Duff	5/29/2015	50.1006	Trem/Act	TR	12	88
TG-DS-48	5/17/2015	1100	Duff	5/28/2015	37.434	Trem/Act	TR	30	70
TG-DS-49	5/17/2015	1130	Duff	5/28/2015	29.2451	Trem/Act	TR	20	80
TG-DS-50	5/16/2015	1200	Duff	6/11/2015	27.7166	Trem/Act	TR	15	85
TG-DS-51	5/17/2015	0900	Duff	6/10/2015	77.8436	Trem/Act	TR	15	85
TG-DS-52	5/17/2015	1030	Duff	6/5/2015	24.7191	Trem/Act	TR	15	85
TG-DS-53	5/17/2015	0930	Duff	6/9/2015	48.3986	Trem/Act	TR	15	85
TG-DS-54	5/17/2015	1000	Duff	6/7/2015	33.9332	Trem/Act/Anthophyllite	TR	15	85
TG-DS-BG	5/26/2015	1400	Duff	5/27/2015	85.2963		ND	12	88
TG-DS-BG	5/26/2015	1400	Duff	6/3/2015	49.1856		ND	12	88
TG-DS-BG	5/26/2015	1400	Duff	6/12/2015	63.5405		ND	10	90
TG-DS-BG	5/26/2015	1400	Duff	6/15/2015	51.3176	Trem/Act	*TR	15	85
TG-DS-BG 5	7/14/2015	1300	Duff	7/14/2015	13.9762		ND	0	100
TG-DS-BG 6	7/14/2015	1300	Duff	7/15/2015	8.7078		ND	TR	100
TG-DS-BG 7	7/14/2015	1300	Duff	7/15/2015	4.487		ND	TR	100
TG-DS-BG 8	7/14/2015	1300	Duff	7/15/2015	5.4125		ND	TR	100
Results verified by Tony Ward 081115.							ND: Not detected		
							TR: trace, <1% Visual Estimate		

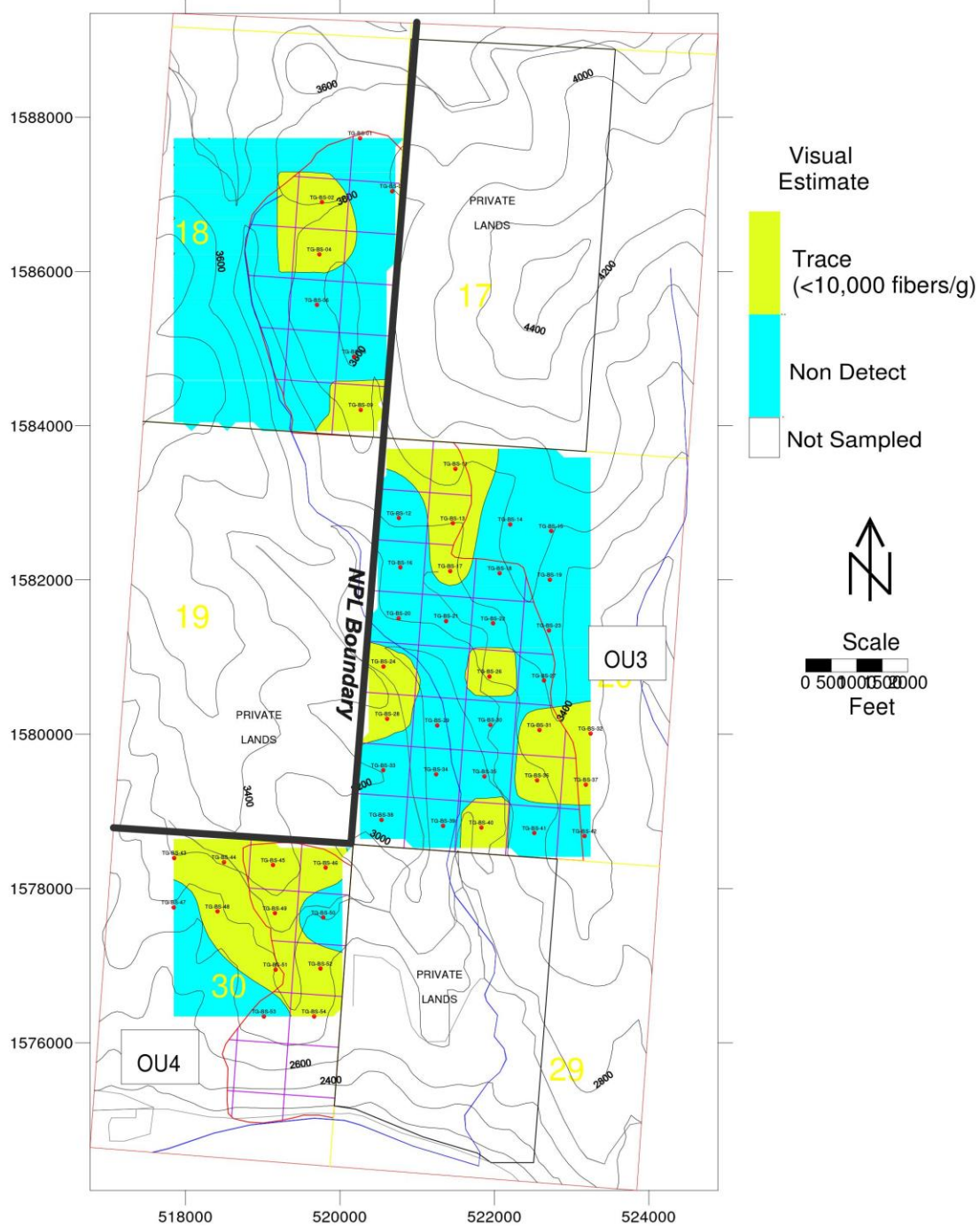
Tubb Gulch - Libby Montana
PLM Duff Screening Results
7/23/2015



PLM Results: Bark

Portage Samples - Bark						
Reservoirs Environmental Inc Results						
August 12, 2015						
			Reservoirs Results			
Sample ID	Northing	Easting	Mineral	Visual Estimate (%)	Non Asbestos Fibrous Components (%)	Non-Fibrous Components (%)
TG-BS-01	1587725.75	520261.71		ND	0	100
TG-BS-02	1586898.30	519768.12	Trem/Act	TR	1	99
TG-BS-03	1587039.71	520676.30		ND	2	98
TG-BS-04	1586222.44	519733.29	Trem/Act	TR	TR	100
TG-BS-06	1585567.12	519701.69		ND	2	98
TG-BS-08	1584896.95	520183.46		ND	1	99
TG-BS-09	1584205.03	520269.14	Trem/Act	TR	5	95
TG-BS-11	1583442.16	521496.39	Trem/Act	TR	0	100
TG-BS-12	1582803.31	520764.43		ND	0	100
TG-BS-13	1582736.06	521461.09	Trem/Act	TR	1	99
TG-BS-14	1582720.48	522205.38		ND	0	100
TG-BS-15	1582635.17	522734.85		ND	TR	100
TG-BS-16	1582162.90	520782.13		ND	3	97
TG-BS-17	1582117.24	521426.86	Trem/Act	TR	TR	100
TG-BS-18	1582088.44	522069.23		ND	2	98
TG-BS-19	1582004.22	522718.14		ND	2	98
TG-BS-20	1581503.82	520759.30		ND	2	98
TG-BS-21	1581468.29	521375.04		ND	0	100
TG-BS-22	1581438.99	521983.57		ND	0	100
TG-BS-23	1581347.33	522706.33		ND	2	98
TG-BS-24	1580878.31	520562.84	Trem/Act	TR	TR	100
TG-BS-26	1580748.73	521935.85	Trem/Act	TR	TR	100
TG-BS-27	1580697.87	522642.74		ND	3	97
TG-BS-28	1580200.58	520612.38	Trem/Act	TR	TR	100
TG-BS-29	1580114.65	521258.52		ND	TR	100
TG-BS-30	1580120.68	521946.53		ND	0	100
TG-BS-31	1580054.46	522583.80	Trem/Act	TR	0	100
TG-BS-32	1580008.30	523245.47	Trem/Act	TR	2	98
TG-BS-33	1579535.17	520561.97		ND	0	100
TG-BS-34	1579483.07	521245.01		ND	TR	100
TG-BS-35	1579454.31	521872.37		ND	0	100
TG-BS-36	1579404.34	522552.60	Trem/Act	TR	0	100
TG-BS-37	1579346.97	523185.39	Trem/Act	TR	TR	100
TG-BS-38	1578889.84	520538.04		ND	1	99
TG-BS-39	1578814.42	521336.64		ND	0	100
TG-BS-40	1578789.85	521831.41	Trem/Act	TR	2	98
TG-BS-41	1578719.81	522517.37		ND	0	100
TG-BS-42	1578681.63	523168.20		ND	1	99
TG-BS-43	1578395.63	517853.99	Trem/Act	TR	2	98
TG-BS-44	1578341.68	518498.61	Trem/Act	TR	2	98
TG-BS-45	1578304.61	519133.52	Trem/Act	TR	4	96
TG-BS-46	1578271.45	519814.86	Trem/Act	TR	TR	100
TG-BS-47	1577755.82	517847.86		ND	TR	100
TG-BS-48	1577703.12	518413.74	Trem/Act	TR	TR	100
TG-BS-49	1577681.04	519156.91	Trem/Act	TR	TR	100
TG-BS-50	1577624.65	519785.02		ND	2	98
TG-BS-51	1576948.56	519165.92	Trem/Act	TR	TR	100
TG-BS-52	1576961.77	519746.39	Trem/Act	TR	0	100
TG-BS-53	1576342.02	519015.86		ND	TR	100
TG-BS-54	1576343.31	519665.32	Trem/Act	TR	TR	100
Results verified by Tony Ward 081115.			TR: trace, <1% Visual Estimate			

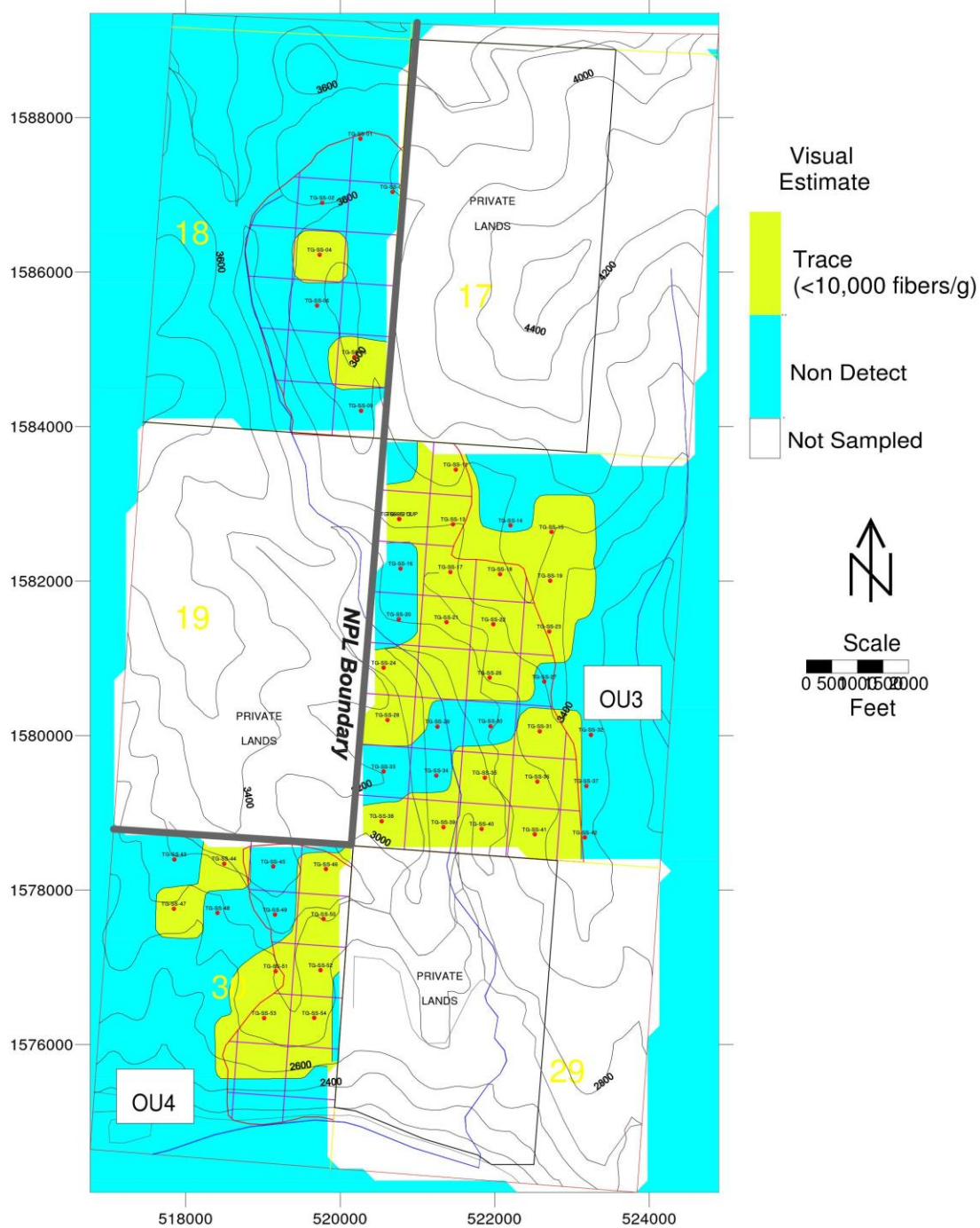
Tubb Gulch - Libby Montana
PLM Bark Screening Results
7/23/2015



PLM Results: Soil

Portage Samples - Soil							
Reservoirs Environmental Inc Results							
August 12, 2015							
				Reservoirs Results			
Sample ID	Portage Collection Date	Portage Collection Time	Media Type	Mineral	Visual Estimate (%)	Non Asbestos Fibrous Components (%)	Non-Fibrous Components (%)
TG-55-BG	5/26/2015	1400	Soil		ND	20	80
TG-55-12	5/20/2015	0730	Soil	Trem/Act	TR	15	85
TG-55-12 DUP	5/20/2015	0730	Soil		ND	12	88
TG-55-16	5/20/2015	0800	Soil		ND	20	80
TG-55-20	5/20/2015	0830	Soil		ND	10	90
TG-55-24	5/20/2015	0800	Soil	Trem/Act	TR	15	85
TG-55-28	5/20/2015	0830	Soil	Trem/Act	TR	12	88
TG-55-33	5/20/2015	0900	Soil		ND	25	75
TG-55-38	5/20/2015	0930	Soil	Trem/Act	TR	15	85
TG-55-11	5/19/2015	0800	Soil	Trem/Act	TR	25	75
TG-55-13	5/19/2015	0730	Soil	Trem/Act	TR	20	80
TG-55-14	5/19/2015	0830	Soil		ND	15	85
TG-55-15	5/19/2015	0900	Soil	Trem/Act	TR	20	80
TG-55-17	5/19/2015	1000	Soil	Trem/Act	TR	20	80
TG-55-18	5/19/2015	1100	Soil	Trem/Act	TR	25	75
TG-55-19	5/19/2015	0930	Soil	Trem/Act	TR	20	80
TG-55-21	5/19/2015	1100	Soil		*ND	15	85
TG-55-22	5/19/2015	1030	Soil	Trem/Act	TR	20	80
TG-55-23	5/19/2015	1000	Soil	Trem/Act	TR	30	70
TG-55-35	5/19/2015	0800	Soil	Trem/Act	TR	25	75
TG-55-40	5/19/2015	0900	Soil	Trem/Act	TR	20	80
TG-55-26	5/18/2015	1130	Soil	Trem/Act	TR	20	80
TG-55-27	5/18/2015	1100	Soil		ND	20	80
TG-55-29	5/18/2015	0830	Soil		ND	15	85
TG-55-30	5/18/2015	0800	Soil		ND	15	85
TG-55-31	5/18/2015	1030	Soil	Trem/Act	TR	15	85
TG-55-32	5/18/2015	1000	Soil		ND	20	80
TG-55-34	5/18/2015	0930	Soil		ND	15	85
TG-55-36	5/18/2015	0800	Soil	Trem/Act	TR	25	75
TG-55-37	5/18/2015	0930	Soil		ND	25	75
TG-55-39	5/18/2015	1030	Soil	Trem/Act	TR	20	80
TG-55-41	5/18/2015	0830	Soil	Trem/Act	TR	12	88
TG-55-42	5/18/2015	0900	Soil		ND	15	85
TG-55-47	5/17/2015	1030	Soil	Trem/Act	TR	10	90
TG-55-49	5/17/2015	1130	Soil		ND	15	85
TG-55-51	5/17/2015	0900	Soil	Trem/Act	TR	10	90
TG-55-52	5/17/2015	1030	Soil	Trem/Act	TR	12	88
TG-55-53	5/17/2015	0930	Soil	Trem/Act	TR	18	82
TG-55-43	5/16/2015	1000	Soil		ND	15	85
TG-55-44	5/16/2015	1030	Soil	Trem/Act	TR	15	85
TG-55-45	5/16/2015	1100	Soil		ND	10	90
TG-55-46	5/16/2015	1130	Soil	Trem/Act	TR	15	85
TG-55-50	5/16/2015	1200	Soil	Trem/Act	TR	15	85
TG-55-08	5/15/2015	1100	Soil	Trem/Act	TR	35	65
TG-55-09	5/15/2015	1000	Soil		ND	35	65
TG-55-02	5/15/2015	1000	Soil		ND	10	90
TG-55-03	5/15/2015	1000	Soil		ND	15	85
TG-55-04	5/15/2015	1000	Soil	Trem/Act	TR	15	85
TG-55-06	5/15/2015	1000	Soil		ND	15	85
TG-55-48	5/15/2015	1000	Soil		ND	10	90
TG-55-54	5/15/2015	1000	Soil		ND	12	88
TG-55-01			Soil		ND	5	95
TG-55-01 Dup			Soil		ND	7	93
				ND: Not detected			
Results verified by Tony Ward 081115.				TR: trace, <1% Visual Estimate			

Tubb Gulch - Libby Montana
PLM Soil Screening Results
7/23/2015



SIGNATURE PAGE

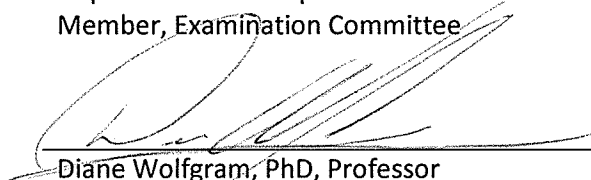
This is to certify that the thesis prepared by Kalli McCloskey entitled "Evaluation of Libby Amphibole Asbestos in Three Media Sources via Polarized Light Microscopy and Transmission Electron Microscopy" has been examined and approved for acceptance by the Department of Industrial Hygiene, Montana Tech of The University of Montana, on this 3rd day of August, 2016.



Julie Hart, PhD, Professor and Department Head
Department of Occupational Health and Safety, Industrial Hygiene
Chair, Examination Committee



Terry Spear, PhD
Department of Occupational Health and Safety, Industrial Hygiene
Member, Examination Committee



Diane Wolfram, PhD, Professor
Department of Geological Engineering
Member, Examination Committee