

Rayonier Mill Off-Property Soil Dioxin Study

Sampling and Quality Assurance Plan

Prepared for



Washington State Department of Ecology
Toxics Cleanup Program
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Abbreviations and Acronyms

ARI	Analytical Resources, Inc.
CDDs	chlorinated dibenzo-p-dioxins
CDFs	chlorinated dibenzofurans
City	City of Port Angeles
DAHP	Washington Department of Archaeology and Historic Preservation
DI	deionized
dioxins	polychlorinated dibenzo-p-dioxins
DOT	Department of Transportation
DQOs	data quality objectives
E & E	Ecology and Environment, Inc.
Ecology	Washington State Department of Ecology
EDD	electronic data deliverable
ESS	Environmental Sampling Supply
FF	focused forested [area]
furans	polychlorinated dibenzofurans
FWEC	Foster Wheeler Environmental Company
GPS	global positioning system
HEC	Herrera Environmental Consultants
IATA	International Air Transport Association
ITT	International Telephone and Telegraph [Corporation]
LCS	laboratory control samples
LEKT	Lower Elwha Klallam Tribe
MBs	method blanks
MDL	method detection limit
Mill	Rayonier Pulp Mill
MS	matrix spike
MTCA	Model Toxics Control Act
NGVD	National Geodetic Vertical Datum
NIST	National Institute for Standards and Technology
ng/kg	nanograms per kilogram
OSRTI	Office of Superfund Remediation and Technology Innovation
PCDD	polychlorinated dibenzodioxin
PCDF	polychlorinated dibenzofuran
PE	performance evaluation
PH-SKC	Public Health - Seattle and King County
PQL	practical quantitation limit
QA	quality assurance
QC	quality control
RD	urban roadside area
RI	rinsate
RP	field replicate
RPD	relative percent difference
RSD	relative standard deviation
SOP	standard operating procedure

Abbreviations and Acronyms (continued)

SQAP	sampling and quality assurance plan
SS	surface soil
SSP	Soil Sampling Plan
TEF	toxic equivalency factor
TEQs	toxicity equivalency concentration
TOC	total organic carbon
USEPA	U.S. Environmental Protection Agency

1.0 Introduction

Ecology and Environment, Inc. (E & E), under contract with the Washington State Department of Ecology (Ecology), has prepared this sampling and quality assurance plan (SQAP) to support the Rayonier Mill Off-Property Dioxin Soil Study, located in Port Angeles, Washington. Herrera Environmental Consultants (HEC), a subcontractor to E & E, also prepared this SQAP and will be working in concert with E & E to execute the activities described herein.

This study focuses on polychlorinated dibenzo-p-dioxins (dioxins) and polychlorinated dibenzofurans (furans) in uplands soils near the former mill. “Off-property” refers to areas outside the current Rayonier Inc. property boundary.

The soil sampling study is based on the design elements discussed in the Soil Sampling Plan (SSP; E & E 2008). The SSP outlines the technical and analytical approaches E & E will employ during the soil sampling field work. This document is a combined field operations work plan and site-specific quality assurance (QA) project plan for field sampling activities. The combined plan, hereafter called the SQAP, includes a brief site summary, project objectives, sampling and analytical procedures, and QA requirements that will be used to obtain valid, representative field samples and measurements. The elements of this SQAP are consistent with the Model Toxics Control Act (MTCA; Chapter 173-340-820 WAC) and Guidelines for Preparing Quality Assurance Project Plans for Environmental Studies (Ecology 2004).

1.1 Purpose

The purpose of this SQAP is to provide the specific methodology for data collection to fulfill the objectives described in the following section and discussed in the SSP (E & E 2008). Work will be performed in cooperation with the City of Port Angeles (City), Lower Elwha Klallam Tribe (LEKT), and other government agencies as required.

1.2 Site Location

The study area is located within Clallam County, Washington within and east of the City of Port Angeles (see Figure 1-1). This study addresses dioxin/furan contamination associated with airborne emissions from the former Rayonier Pulp Mill (Mill).

The proposed study area surrounds the former Mill, extending to Tumwater Creek on the west, to Buchanan Drive on the east, and to Lauridsen Boulevard on the west side of the southern border (see Figure 1-2). The east side of the southern border extends approximately one mile inland from the bluff to include the Drennan-Ford Funeral Home and Crematory. The study area encompasses approximately 4.2 square miles. In addition, three targeted sample transects will run north-south, extending south of the primary study area (see Figure 1-2).

Based on the judgment of field sampling personnel, flexibility will be allowed to collect some samples slightly beyond the mapped boundary of the primary study area if that will provide more favorable sampling locations, such as mature and relatively undisturbed forested locations.

1.3 Site History

The Olympics Forest Products Company constructed a pulp mill along the waterfront in 1930. The mill later merged with two independent companies in 1937 to become Rayonier, Inc. In 1968, International Telephone and Telegraph (ITT) Corporation purchased Rayonier, Inc., renaming the mill ITT Rayonier. The mill operated under ITT Corporation until 1994, when the mill was spun off from ITT Corporation and resumed operating under the name Rayonier, Inc. until its closure in 1997. Descriptions of Mill history and operations are provided by Foster Wheeler Environmental Company (FWEC; 1997) and Integral Consulting Inc. (2006).

The Rayonier property, which has been almost completely cleared of its mill facility and outbuildings, totals 80 acres. The property is located in Section 11 of Township 30 north, Range 6 west, at a latitude of 48° 07' 00" north and longitude of 123° 24' 25" west. Most of the property extends into the eastern portion of Port Angeles harbor. The northern portion of the property is generally flat, with relatively steep bluffs rising rapidly to approximately 75 feet above National Geodetic Vertical Datum (NGVD) immediately to the southeast and southwest (HLA 1993). The terrain continues to rise to approximately 200, 265, and 150 feet above NGVD within approximately one mile southeast, south, and southwest of the property, respectively.

Throughout the Mill's operating history, air emissions were released from numerous sources on-site under normal operating conditions, including the recovery and hog fuel boiler stacks, the chlorine dioxide generator, and vents in the bleach plant, acid plant, and blowpits. The recovery boiler was constructed in 1974, in part to reduce sulfur dioxide emissions. At the same time, scrubbers and demisters were placed on the recovery boiler stack (U.S. EPA 1993). A scrubber and demisters also were installed on hog fuel boiler No. 6 in 1974 (FWEC 1997).

Rayonier used wood chips, including salt-laden wood, in the on-site hog fuel boiler (Integral 2006). Due to the location of the Mill on Port Angeles harbor and the abundance of wood as a source of fuel for on-site burners, the Mill burned wood chips and wood wastes coming from logs floated in Port Angeles harbor. Use of salt-laden wood in hog fuel burners can result in significantly higher emissions of dioxins/furans than burning salt-free wood (Duo and Leclerc 2004; Lavric et al. 2004; Luthe et al. 1997; Luthe et al. 1998; Pandompatam et al. 1997; Preto et al. 2005; Uloth et al. 2005). Combustion of salt-laden hog fuels in the hog fuel boilers is considered the primary source of dioxins/furans emitted from the former Mill. Also, incineration of water treatment system sludge may have been a source of dioxin/furan emissions from the hog fuel boiler.

Limited testing was performed in 1988 on samples collected from hog fuel boiler No. 6 at the former Rayonier Mill, including bag house ash (1,310 nanograms per kilogram [ng/kg] 2,3,7,8-TCDD TEQ) and washed ash (170 ng/kg 2,3,7,8-TCDD TEQ; FWEC 1997). The presence of dioxins/furans has been confirmed in further sampling performed on samples from the hog fuel boiler. Samples from the hog fuel boiler were obtained in 1989, with analytical results documenting total dioxin and total furan concentrations of 2,700 ng/kg and 19,000 ng/kg in boiler ash and 22,000 ng/kg and 22,000 ng/kg in filter ash, respectively (FWEC 1997). In 1995, stack tests of air emissions from the hog fuel boiler confirmed the presence of dioxins/furans (FWEC 1997).

Additional samples of bag house fly ash (total TCDD 160,000 ng/kg; total TCDF 64,000 ng/kg) and filter ash (total TCDD 380,000 ng/kg and total TCDF 33,000 ng/kg) were collected in 1991 and 1993, respectively. In 1996, concentrations of 2,3,7,8-TCDD (110 ng/kg) and 2,3,7,8-TCDF (350 ng/kg) were detected in vacuum filter ash (FWEC 1997). Generally, dioxin/furan loading is associated with fly ash as opposed to grate or filter ash (Yake et al 1998). A complete description of these sample results is provided by FWEC (1997) and Integral Consulting Inc. (2006).

1.4 Objectives

The goal of the Rayonier Mill Off-Property Soil Dioxin Study is to provide an increased understanding of dioxin/furan soil contamination in areas surrounding the former Rayonier Mill, including the magnitude and likely sources for contamination of surface soils. Specific study objectives include:

- Determine the magnitude of dioxin/furan contamination in off-property surface soils potentially impacted by airborne emissions from the former Rayonier Mill, and
- Determine the relative contribution to measured soil dioxin/furan concentrations of former Rayonier Mill emissions compared to other potential sources.

The study design must meet a resource constraint that will support the collection and analysis of not more than 100 soil samples. Based on discussions with Ecology, time constraints also led to a decision that the study design rely on a single mobilization for sample collection rather than a phased sample collection approach where study designs could be refined based on initial, early-phase results.

This study is not intended to delineate the full extent or boundary of Mill-related impacts, even though it will include sampling of a relatively large area surrounding the former Mill. The collected data also will not support interpreted property-by-property mapping of soil dioxin/furan concentrations, nor will the sampling be sufficiently detailed to characterize potential exposures for risk assessment purposes. Finally, this study does not focus on characterization of natural or anthropogenic background values for dioxins/furans in soil.

1.5 Overview of Study Design

The SSP provides a detailed discussion of the study design. A relatively large study area was defined for this study, based on air modeling, an odor study, location of existing dioxin/furan sources, and other factors. Multiple exclusion and/or preference criteria were applied at various stages of the development of the sampling design to identify sampling locations deemed most appropriate to meet the study objectives.

To meet the objective of sampling at locations reflecting the upper-range of concentrations in soil concentrations, preference is given to sampling in wooded areas not otherwise excluded (e.g., by steep slopes). Previous studies and models for air particulate contaminants support a conclusion that soil dioxin/furan concentrations are generally higher in forested areas than in open areas. Since available wooded areas are limited within the study area, many samples will be collected from developed properties.

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Sampling zones are defined primarily as a means to apply a sample allocation scheme in which sampling densities are varied across the study area. The sampling zones also reflect some differences in available land use and land cover types across the study area; however, all other aspects of sample collection (compositing and sampled depth interval) are identical across all sampling zones. The defined sampling zone boundaries follow topographic contours or transportation routes as a matter of convenience and are not assumed to precisely define areas of varying dioxin/furan concentration. Because very limited information was available to use as a basis for defining the spatial patterns of soil dioxin/furan contamination, more detailed and complex approaches to providing a sample allocation with varying densities across the study area were deemed unnecessary.

All samples will be collected from a single, uppermost depth interval of 0 to 3 inches and will be composites of a small number of subsamples. All samples will be analyzed for seventeen 2,3,7,8-substituted congeners and their ten homologue groups, as well as total organic carbon (TOC).

After receipt of the validated data, the first step in data evaluation is to explore the dataset using summary and descriptive statistics to determine the most appropriate steps for further data analysis. Evaluation of chemical data for pattern recognition, modeling, and statistics is referred to as “chemometrics.” An expert in chemometrics, Dr. Scott Ramos of Infometrix, Inc., has been retained to perform the data evaluation and source identification analyses.

Once the analytical results have been reported to study participants and the data evaluation process is complete, a draft final report will be developed and presented for public review.

2.0 Project Team and Schedule

The following sections summarize project staffing and the proposed schedule for activities described in this SQAP. This study includes the following subtasks:

- Project planning and agency coordination
- Property identification and access coordination
- Mobilization
- Soil sample collection
- Laboratory preparation and analysis
- QA/QC Management
- Data reporting

2.1 Project Planning and Coordination

Ms. Connie Groven, Ecology project manager, will provide overall project coordination, supply government-furnished data and services, provide review comments on the report, and coordinate with E & E. Ms. Alma Feldpausch, E & E project manager will be responsible for executing the approved SQAP, overseeing the collection and storage of field samples, and reporting analytical results to Ecology.

2.2 Property Identification and Access Coordination

Ms. Feldpausch will obtain access agreements from participating property owners in consultation with Ecology. This will include developing a database of property owners within the study area using existing information available from the City and Clallam County tax assessor offices, generating an interview used to screen candidate property owners, conducting door-to-door recruiting activities, as necessary, and making appointments with selected property owners for sample collection. Ms. Groven will review and approve all materials used in the recruitment process (access agreement, recruitment letters) and will provide informational materials to be sent to candidate property owners.

2.3 Mobilization

Ms. Feldpausch and HEC field team leader, Mr. Bruce Carpenter, will manage the mobilization to and from the site. Mobilization will include the following activities:

- Procurement of subcontractor services, equipment and materials - This may include, but is not limited to, field office rental, laboratory services, and waste disposal and safety supplies.
- Coordination with the City, LEKT, and Washington Department of Archaeology and Historic Preservation (DAHP) - Sampling will require 24-hours advance notice to City and LEKT representatives, as well as notice to the DAHP.
- Coordination with property owners – All property owners from whom signed access agreements have been obtained must be contacted prior to sampling to confirm appointments.

2.4 Soil Sample Collection

Ms. Feldpausch and Mr. Carpenter will be the consultant field managers and will be responsible for the health and safety of the field crew, collection of soil samples in accordance with the SQAP, and transport of samples to the analytical laboratory for chemical analysis. Health and safety of the field staff is discussed in the site-specific health and safety plan, provided as Appendix A. Ms. Feldpausch will coordinate with the property owners for this project and Mr. Carpenter will ensure accurate positioning for sample locations.

Ms. Sandra Pentney, registered professional archaeologist with E & E, will be responsible for developing the cultural resource monitoring and reporting protocol in consultation with the City, LEKT, and DAHP. She, along with an assistant archaeologist, will monitor the soil sampling process in the field according to the procedures provided in the protocol.

2.5 Laboratory Preparation and Analysis

Under the direction of the consultant field managers, E & E and HEC personnel will be responsible for the visual description of soil samples, sample processing, and delivery of samples to the analytical laboratory. Established protocols for decontamination, sample preservation, holding times, and chain-of-custody documentation will be observed.

Ms. Angelica Whetung, Axys Analytical Services, Ltd., will be responsible for dioxin/furan analyses of the collected samples using the approved methods described in Section 6. Ms. Susan Duniho, Analytical Resources, Inc. (ARI), will be responsible for TOC analysis using the methods described in Section 6. The laboratory representatives will handle and analyze the submitted samples in accordance with analytical testing protocols and QA/QC requirements described in Sections 6 and 7, respectively. A written report of analytical results and QA/QC procedures will be prepared by the analytical laboratory and included as an appendix in the data report.

2.6 QA/QC Management

Mr. David Ikeda, E & E, and/or Ms. Gina Catarra, HEC, will perform quality assurance oversight for the laboratory programs. They will ensure that the laboratory analytical and QA/QC data are considered valid and procedures meet the required analytical quality control limits. They

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will oversee the data validation of the analytical chemistry results, which will be performed by EcoChem, Inc.

Ms. Ann Bailey, EcoChem, Inc., will be responsible for validation of the analytical data provided by Axys, using the methods described in Section 7. A written report of data validation results will be prepared by EcoChem along with an electronic database containing all laboratory data and validation notes. This report will be included as an appendix in the data report.

2.7 Data Report

Ms. Feldpausch will coordinate the preparation of a data report, consisting of a technical memorandum, for submittal to Ecology. Following this report, a final project report will be generated that will include the results of the chemometric data analyses.

2.8 Schedule

A schedule for sampling, including planning, subcontract procurement, field activities and mobilization/demobilization, and reporting is provided below. A revised schedule, if necessary, will be provided to Ecology following approval of the SQAP and notice to proceed.

Task	Date
Analytical laboratory and data validation subcontract acquisition	May – June 2008
Field planning	July – August 2008
Property access coordination	July – August 2008
Field mobilization	September 3, 2008
Field sampling	September 4 – 16, 2008
Field demobilization	September 17, 2008
Sample analysis	September 4 – October 31, 2008 (45-day analysis period)
Data validation	October 6 – December 5, 2008 (30-day evaluation period)
Reporting – Technical Memorandum	December 2008 – February 2009
Reporting – Final Project Report	February 2009 – June 2009

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3.0 Sampling Strategy – Property Identification and Access

The following field activities for this investigation were determined based on the objectives outlined in Section 1.4 of this SQAP:

- Property access coordination, and
- Collection of 100 investigative surface soil samples and five replicate samples.

Property access is addressed in this section while soil sample collection is addressed in Section 4. An overview of the process for property identification and acquisition of the access agreement is presented in Figure 3-1.

3.1 Identify Candidate Properties

The objective of this task is to identify potential properties within the study area that meet criteria for sample collection. The criteria will identify properties that have minimally disturbed soils that likely represent accumulation of dioxin/furan emissions deposition.

During the sample location identification process, the properties will be assigned a tier level with Tier 1 properties being the most ideal for sampling and Tier 3 being least ideal but acceptable in the event that no other properties are available within a given cell. Tier E properties include properties that are excluded due to recent development, soil disturbance, or other confounding factors, or may include properties excluded from consideration at the request of the property owner. The tiers to which properties within the study area will be assigned include:

- Tier 1: Undeveloped property of at least one acre open space or forest that appears to be at least 30 years old.
- Tier 2: Partially developed property (e.g., residential or commercial and open field) with improvements constructed prior to 1977 and forested area that appears to be at least 30 years old or undeveloped property less than one acre in size.
- Tier 3: Developed property (e.g., residential or commercial) with exposed soil or vegetative land cover and buildings constructed prior to 1977.
- Tier E: Excluded properties that have been developed or otherwise disturbed since 1977.

The first step in identifying candidate properties involves developing a list of property owners within the study area. This information will be obtained from the City and Clallam County planning and tax assessor offices and will include property tax identification number, age of structures (if any), property street address, property owner(s) name, and mailing address and phone number of property owner(s), if available.

The second step in identifying candidate properties involves inspecting historical and current aerial photographs of the sampling area to evaluate the level of development and time-frame of observed development. The age of all existing structures (e.g., homes, businesses, outbuildings), as provided by government records, also will be reviewed to identify developed properties with structures built prior to 1977.

At this time, properties are assigned to a tier level but may be re-assigned to a different tier level as additional information is gathered for each property. Properties that have no to low development likely will be assigned to Tier 1 or Tier 2 and properties that are developed but have grassy or other open areas, such as lawns, likely will be assigned to Tier 3. Properties that are entirely paved or were recently developed will be assigned to Tier E.

3.2 Contact Property Owners

The third step in the process for identifying sample locations involves requesting written permission from select property owners to collect surface soil samples. Tenants who are not property owners may not grant access to the property for sample collection. Access agreements will be sent to select owners of properties classified as Tier 1 through Tier 3, with contact of multiple property owners within a given grid cell or transect area as described below. The access agreement request letter and access agreement are included in Appendix B.

Grid Cell Locations

A minimum of three property owners will be contacted per grid cell (if three distinct property owners exist per grid cell). Irregular shaped grid cells along the zone boundaries will be combined with adjacent cells to the extent necessary to maintain the allotted sample size and desired spatial coverage. Where building age information is available for developed properties, the properties within a given grid cell with the longest history since last development will be sent an access request letter. For developed properties, additional properties representing subsequent decades since development also will be sent letters. For undeveloped properties, those with the oldest and/or largest stands of trees (or when no trees are present, those with the largest undeveloped area) will be sent access request letters.

The properties will be selected first from properties classified as Tier 1, then Tiers 2 and 3 as necessary. When properties cannot be differentiated based on age of structure (if any structure is present) or undeveloped surface area, an attempt will be made to distinguish those that are located further from high traffic areas and railways, adjacent to forested areas, or have larger undeveloped areas in the “back” yard. Time since last sale also may be a factor in selecting properties, with properties that have been more recently sold being less desirable as they may have been landscaped prior to the sale or less may be known about the property history by newer owners. Other determinations may be made based on best professional judgment.

Targeted Sample Locations

For the upslope targeted transect sampling locations, a minimum of nine property owners within each transect area will be contacted and sent access request letters. For the targeted Highway 101 sample locations, two locations will be selected from an area representing a location where

increased emissions may be expected due to automobiles laboring uphill. Samples likely will be collected from a median or right-of-way within 10 feet of the highway shoulder but will not be collected from a drainage ditch. Targeted forested locations will be selected based on size of the forested area and age of trees. Preference will be given to larger and older stands of trees. Ideally, forested sample locations will be at least five acres in size and trees will be no less than 30 years old, as determined by visual observation and property owner information.

For all recruitment efforts, loss of some sampling locations and re-classification of a property to Tier E is expected because access is not granted by the property owner or because exclusion criteria result in elimination of the sampling location. Ideally, more than one property will be available within a grid cell or transect area for sample collection.

The access agreement will address the legality of entering private and public property, and will provide a check box where property owners can elect to receive a copy of the results from the samples collected from their property. The access agreement will state that copies of the results will be mailed to those requesting a copy prior to any public release of information. The results of the study and all paperwork related to the sampling of properties will become part of the public record. This will include log books, field forms, and interview results that include property owner and tenant names, contact information, sample coordinates, and other information gathered during the recruitment and sampling processes. However, no names or addresses of property owners will be printed in the final reports. Sample location coordinates will be captured in Ecology's EIM database and sample locations will be displayed in report figures.

Access agreement letters sent to property owners also will explain that if an owner grants permission to have their property sampled, they will be asked questions regarding the property development history and possible influence of other dioxin/furan sources on their property to determine if the soils are likely to represent long-term accumulation of emissions deposition from the Mill.

3.3 Screen Candidate Properties

The fourth step involves contacting owners who have granted permission to sample their property to conduct a screening interview. The interview will be conducted at the earliest possible time following written receipt of the signed access agreement. The interview will be conducted over the telephone and will last approximately 15 minutes. Soil disturbances since 1977 or influence from localized sources of dioxins/furans (e.g., use of pentachlorophenol-treated products, historical application of DDT, house fires, etc) will result in reassignment of the property to Tier E. In addition, property owners will be asked the age of the house, when and if there has been any landscaping, and if there was an underground sprinkler system installed. An example of the property owner and tenant interview questions are provided in Appendix B.

Based on information gained during the interview with the property owner, the property will be re-classified as a Tier E property if one or more of the following conditions are met:

- Properties with homes built after 1977.

- The property is located 150 feet of Highway 101 due to potential application of herbicides and deposition of vehicle exhaust along the roadway.
- The property is located within 150 feet of a railway corridor due to potential application of herbicides and deposition of locomotive exhaust within the corridor.
- The property is adjacent to a hazardous waste or other contaminated sites identified on regulatory databases and under investigation by regulatory agencies such as EPA or Ecology.
- The property is adjacent to a heavily industrialized area or landfills.
- The property is an area where soil erosion or deposition from off-property areas is likely to occur, including steep slopes, riparian wetlands, or floodplains.

If an owner does not wish to participate in the study, the property will be re-classified to Tier E. Owners who are interested in participating in the study will be screened using the interview described above. Properties not excluded following the interview process will be considered candidate sample locations. Throughout the recruiting process, the spatial distribution of candidate sample properties will be assessed to determine where spatial coverage may be lacking. Areas where candidate properties have not been identified will be the subject of more intensive recruiting efforts.

Areas lacking candidate properties will be visited by E & E staff in an attempt to obtain permission to sample. Two staff will visit Port Angeles at the earliest practical time to request access to the property for sampling. Attempts to visit the property or property owner will be recorded in a log. If property owners grant permission to be considered in the study, then the screening interview will be conducted at that time to determine if the soil disturbance history or presence of dioxin/furan sources suggest that the property is not a suitable soil sampling location. If a property owner does not wish to participate in the study, the property will be re-classified as a Tier E property.

3.4 Select Properties for Sampling

The fifth step of the recruitment process involves selecting the property to be sampled for each grid cell or transect area from among those properties for which a signed access agreement has been obtained. One property per grid cell will be selected for sample collection from those candidate properties which have not been eliminated based on information obtained during the interview. Three properties will be selected within each of the three targeted transect areas and two targeted highway samples will be selected along Highway 101. In addition, ten forested areas will be selected from Zones E2 and E4.

When more than one suitable candidate property has been identified within a grid cell or transect area, the Tier 1 property with the longest history without development or influence from non-Mill dioxin/furan sources will be selected as the sample location for a particular grid cell or targeted sample area. If no Tier 1 properties are present within a grid cell or targeted sample area, the Tier 2 property with the longest history without development or influence from non-

Mill dioxin/furan sources will be selected as the sample location. Tier 3 properties then will be considered if no Tier 2 properties are available. For the zoned sample areas, if there are a greater number of grid cells per zone than there are allocated samples per zone then the irregularly-shaped cells drawn along the zone boundaries will be combined with adjacent cells while maintaining the allotted sample count per zone and desired grid spacing.

In the event that no eligible properties are available and/or no property owners grant permission to collect samples within a grid cell or transect area, assistance will be requested of Ecology to recruit participants and/or re-allocate the sample to an alternate grid or targeted sample area.

3.5 Schedule Sample Collection Activities

Properties selected for sampling will be designated as sampling locations and the owners will be notified via their preferred mode of contact of the intent to sample. At this time, E & E will coordinate access to the property and will schedule an approximate date and time between September 4 and September 13 to collect the sample. E & E will explain the process for sample collection and answer questions owners may have regarding the sample and analysis process. Owners and/or tenants will be requested not to water their lawns for three days prior to the scheduled sample date and will be asked to restrain outdoor pets during the scheduled sample time.

Owners will be contacted one to two days prior to the scheduled sample day to remind owners of the appointment. Owners do not need to be present at the time of sampling provided the access is not restricted by security fencing or other obstruction.

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4.0 Sampling Strategy – Soil Sample Collection

This section addresses soil sample collection methods, including sample location, equipment/material requirements, decontamination of sampling equipment, sample container labeling, and field documentation. Approximately 100 investigative surface soil samples will be collected throughout the study area.

4.1 Property-specific Sample Location Procedures

The following procedures should be used to determine surface soil sample locations for dioxin/furan and TOC analyses. A field sampling form has been developed specifically for this project and it is provided in Appendix C. Forms will be filled out during the site location selection process and provided to each sampling team. The information on the form will be used by the sampling team to select specific sampling points.

4.1.1 Selection of Sample Locations

For the purposes of this SQAP, sampling location refers to the general site or property where a sample will be collected and sampling point refers to the area within a sampling location that a soil sample will be collected. Each sampling point will be represented by five subsamples. A discussion of sample location selection rationale is provided in Section 3 of the SQAP.

4.1.2 Surface Soil Sampling Points

Surface soil sampling points will be selected from the least disturbed portions of the sample locations throughout the study area. The sampling point should represent a consistent set of depositional and land use conditions at each sampling location. The following areas will be excluded when selecting sampling points:

- Disturbed areas at developed sites (i.e., construction sites, areas around concrete pads or foundations, telephone and electric poles, landscaping and ornamental planters, building drip lines, down spouts, gardens, dog runs, and areas of animal burrowing activity). Generally, soil will not be collected within six feet of disturbed areas though this distance may be increased based on field conditions.
- Areas near wooden structures where treated wood may have been used. Soil will not be collected within a 20-foot radius of creosote-treated wood; a six-foot radius from other forms of treated wood will be excluded from sampling.
- High-traffic areas (i.e., parking lots, roadways, sidewalks). Soil will not be collected within six feet of high-traffic areas.
- Burn pits, fire pits, and areas adjacent to chimneys of wood burning fireplaces. Soil will not be collected within 20 feet of burn pits, fire pits, and other incineration sources.

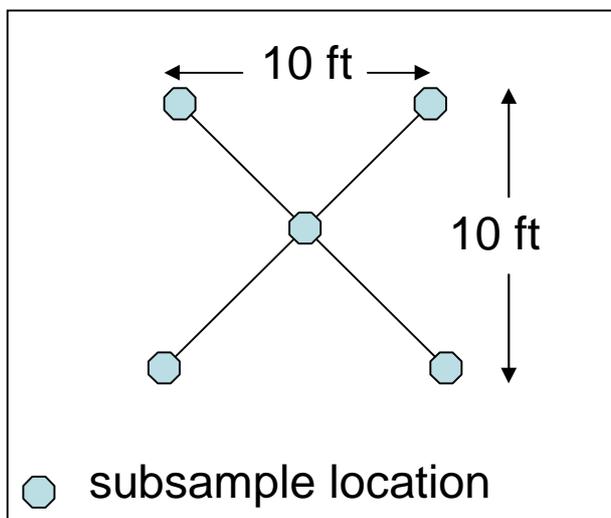
- Steeply sloped areas or areas potentially shielded from deposition of emissions from the Rayonier mill will be avoided.
- Hummocks, paths used by animals or humans, areas lacking ground cover, and other areas disturbed by treefall or animal digging within forested areas will be avoided.
- Forested areas dominated by immature trees less than 30 years old, areas that were recently reforested, and areas where there is evidence of recent fires will be avoided.
- The transitional area between forested and undeveloped properties where dumping is more likely to occur will be avoided.

If samples are collected in the vicinity of these exclusion areas, the presence of, type, and distance from the exclusion area will be documented in the field log book and/or field sampling forms and photographs.

4.1.3 Subsample Locations at Sampling Points

After selecting a sampling point at a property, five subsample locations will be established and marked on the ground using pin flags. A default design for collecting subsamples will be used as a point of departure for modification by field personnel using their best judgment on collecting representative samples. Collectively, the set of subsamples should represent consistent conditions across the sampling point.

The default design will be to collect subsamples from the four corners and the center of 10-foot by 10-foot template (see example below). Most residential yards will accommodate this template size. Within larger yards, the standard template can be used at any representative subarea as determined by the field personnel. At residential or other developed properties, this layout may be modified as long as subsamples are separated by at least 5 feet. If this is not possible, an alternate property should be considered for sampling. For sampling at forested locations, the size of the sampling template may be modified upward from a minimum 10-foot by 10-foot square; samples may be collected up to 50 feet apart, and slightly off of the corners of a square template if obstacles or excluded ground surfaces occur.



After marking subsample locations with pin flags, a photograph of the sampling point will be taken and recorded in the field log book. A site sketch of landmarks on the property, the sample location point, and other pertinent information will be recorded on the field sampling form (Appendix C). In addition, field staff will record the latitude and longitude at the center of the sampling point using a global positioning system (GPS) device.

4.2 Sampling Equipment/Materials Required

The following list of equipment will be used during the soil sample collection process.

- Field copy of the SQAP
- Map of the study area and planned sampling locations
- Field Logbook
- Field Sampling Forms
- Pens with indelible ink
- Nitrile gloves
- Pin flags
- Digital camera
- GPS unit
- Tape measure (at least 100 feet long) and compass
- Ruler
- Spade shovel
- Stainless steel bowls
- Stainless steel spoons
- Plastic sheeting (for surface vegetative layer and excavated soils)
- Aluminum foil (to cover stainless steel bowl while subsamples are collected and to place in bottom of hole to capture fine-grained material while scraping sidewall)
- Sample containers (provided by laboratory: 8-oz and 4-oz glass, pre-cleaned with Teflon lids)
- Sample labels

- Clear plastic packing tape (to tape over sample labels)
- Strapping or duct tape (to tape up coolers)
- Chain-of-Custody Forms
- Cooler(s)
- Custody seals
- Bubble wrap
- Prepaid Federal Express labels for coolers from analytical laboratory
- Ice for coolers
- Plastic bags (gallon size zip lock for ice and chain-of-custody)
- Plastic bags (quart size zip lock for sample containers)
- Plastic trash bags (lawn and leaf)
- Decontamination supplies: 20-liter buckets with lids, stiff bristle brush, spray bottles, Liquinox™ or Alconox™ non-phosphate detergent
- Deionized (DI) or distilled water
- Paper towels
- Personal protective equipment (safety glasses with side shields, steel toed boots, sun screen, hat)
- Potting soil for use in site restoration

4.3 Sample Collection Procedures

The following procedures should be used to collect surface soil samples for dioxin/furan and TOC analyses. Note that because cigarette smoke is a potential source of dioxins/furans, there shall be absolutely no smoking at any time during the sample collection process. Exhaust from vehicles and electrical generators can also be a source of dioxins/furans and therefore sample collection shall be performed away from running vehicles or generators (all combustion engines, to the extent possible). Also, note that soil samples will not be sieved although the analysis of bulk samples is inconsistent with MTCA (see E & E 2008 for further discussion).

4.3.1 Pre-Sample Collection Activities

Place bubble wrap on the bottom of the sample cooler. Place one garbage bag (lawn and leaf size) in the sample cooler on top of the bubble wrap. Divide one bag of ice into several (3 or 4)

double bagged gallon-size zip lock bags and place them inside the garbage bag at the bottom of the sample cooler. The garbage bag will help to ensure that water from the ice does not leak out of the cooler during shipping. Place the temperature blank in the cooler with the ice. Samples should be placed in the cooler upon collection.

4.3.2 Surface Soil Sample Collection

Note location and current site conditions in field books and on field sampling form. Record sampling data on field sampling form and in field book per section 4.7. Establish subsample locations with pin flags per section 4.1.3. Place plastic sheeting in area of sample location to stockpile groundcover and excavated soil. Materials placed on plastic sheeting will be used to back-fill and re-cover sampling locations.

Remove Groundcover

Groundcover may consist of grass, other vegetation, or rocks/pebbles. An area of approximately 8 inches by 8 inches will need to be uncovered; this can be measured using a ruler. The actual area may vary by site depending on how rocky the soil is and how much vegetation is present.

- Remove the surface layer of grass, leaves, or twigs at each subsample point using a spade. The groundcover should only be removed to the point where soil is exposed, being careful not to disturb the soil below.
- In forested areas, forest litter (undecomposed identifiable dead plant material) and forest duff (partially decomposed organic material) will be removed using a spade. Scraping the sidewall during sample collection will include the humus horizon (completely decomposed organic material) that underlies the forest duff.
- If the sampling point does not contain vegetation, then any rocks or pebbles can be brushed aside by the sampler(s) using a gloved hand.

Subsample Collection

Once the vegetation and/or rocks/pebbles have been removed, collect surface soil subsamples as follows:

- Put on a clean pair of nitrile gloves.
- Excavate the hole to a depth of 3 inches with a clean spoon.
- Remove roots and large cobbles or other objects that would impede the spoon from moving continuously along the sidewall.
- Place a clean piece of aluminum foil at the bottom of the hole.
- Scrape the side of the hole beginning at the bottom with the spoon. Scrape slowly adjusting pressure of the spoon against the sidewall to collect an equal amount of soil

Rayonier Mill Off-Property Soil Dioxin Study SQAP

along the entire 3-inch sidewall length. More pressure will be required to scrape hardpan material and less for unconsolidated sandy material.

- Empty the spoon and the soil on the foil into an 8-ounce glass jar.
- Scrape the sidewall multiple times if necessary to fill the jar.
- Empty the soil from the jar into a stainless steel bowl.
- Repeat this process at the four other subsample locations.
- Remove any large rocks or large fragments of organic matter such as sticks or roots from the bowl, taking care to retain soil particles adhered to debris to the extent practical.
- Homogenize the soil in the bowl by mixing with the collection spoon and then separate the soil into four equal aliquots by drawing an “X” in the soil with the spoon.
- Place one spoonful of soil from each quarter into a clean sample container provided by the laboratory, alternating containers, and continuing until both containers (one 4-ounce and one 8-ounce jar) are full.
- Place the labeled sample containers into an iced cooler.
- Provide the remaining sampled soil for split sample collection.
- Remove pin flags once soil samples have been collected and return site to original state as best as possible. Potting soil may be used to fill any holes created by sample removal.
- To collect replicate soil samples, fill two glass jars at each subsample location, homogenize the soil and fill four sample containers (of each 4-ounce and 8-ounce size) following the same procedure listed above for two containers.

General Sample Collection Procedures

- Sample labels and handling are discussed in Section 5.
- A clean stainless steel sampling bowl, measuring cup, and spoon will be used at each sample location.
- The aluminum foil covering the stainless steel bowl will be disposed of after sample collection.
- Remove and dispose of gloves after collection from each sampling point.
- Remove pin flags once soil samples have been collected and return site to original state as best as possible. After collection of the split sample, return leftover soil in the mixing bowl to the hole; potting soil may be used to fill any holes created by sample removal.

4.4 Decontamination Procedures

The stainless steel spoons, measuring cups, and mixing bowls used to collect samples will be dedicated, single-use equipment and will not need to be decontaminated in the field. The single-use sampling equipment will be decontaminated and certified clean by Environmental Sampling Supply (ESS), according to "Procedure 1." Procedure 1 is recommended for equipment used to collect samples submitted for extractable organic and pesticide analysis. This procedure includes the following steps:

- Washing the equipment with a non-phosphate detergent.
- Multiple tap water and ASTM Type I deionized water rinses.
- 1:1 nitric acid rinse.
- n-hexane solvent rinse.
- Oven drying the equipment.
- Wrapping all equipment in sterile aluminum foil.

The cleaned equipment will be provided with a "Certificate of Compliance," noting that the equipment is pre-cleaned certified by ESS. After use for sampling, the equipment will undergo decontamination as described below and will not be used again for this sampling effort.

All reusable or nondedicated field equipment (e.g., the spade/shovel) will be decontaminated prior to reuse but will not be decontaminated between subsample collection at one location. Decontamination of nondedicated equipment before reuse will be conducted to avoid cross-contamination between samples and to protect the health and safety of the field sampler(s). The following decontamination sequence should be used for the spade/shovel prior to collecting the first sample and between each use:

- Nitrile gloves (or equivalent) must be worn during decontamination.
- Rinse with potable water, collecting rinse water in one of the decontamination buckets.
- Wash with a spray bottle containing Liquinox™ (or equivalent nonphosphate detergent) and water and clean with the stiff-bristle brush until all evidence of soil or other material has been removed.
- Rinse with DI or distilled water three times, ensuring that all soap from the previous step has been removed.
- Place the spade/shovel on a piece of aluminum foil to air dry; decontamination has been completed.
- A trash bag should be provided for waste paper towels, aluminum foil, and used nitrile gloves.

4.5 Sample Designation

Samples will be designated by a seven-digit alphanumeric system referencing the sample area and sample number within the location. For example, “W101SS” denotes the surface soil sample collected from cell 01 within zone W1. The sample nomenclature is discussed further in Section 5.

4.6 Sample Container Labeling

Sample handling is discussed in Section 5.0.

- Each sample container must have a sample label affixed to the outside of the container in an obvious location. Information must be recorded using a permanent marker.
- The label must include project name, sample identification, and analysis. If possible, this information should be filled in before sample labels are sent to the field.
- Immediately after sampling, record the date and time (military time [i.e., 1330]) of sampling along with the initials of the sampler(s) on the sample label.
- The completed sample label must be taped over with clear tape (i.e. packing tape) to prevent the label from getting wet, smudged, or lost during transport.

4.7 Field Documentation

4.7.1 Field Log Books

Field logbooks are intended to provide sufficient information to reconstruct events that occurred during field activities. The following are examples of information to be included by the sampler(s) in a field logbook:

- Project name and location.
- Name, date, and time of entry.
- Names and responsibilities of field crew members
- Name and titles of any site visitors involved in or actively observing the sampling.
- Descriptions of deviations from the sampling procedures and any problems encountered.
- Weather information including air temperature and recent precipitation.
- Date and time of sample collection.
- General observations, including the setting / features surrounding the sampling location, topography, etc.

- Start and stop times of work.

All available space on a page must be filled; if blank space exists after the last entry on a page, then a line should be drawn through it and the initials of the documenter provided with a date.

4.7.2 Photographs

Photographs will be taken at each sampling location and recorded on the field sampling form (Appendix C). Photographs will be taken to document field conditions, including the features and structures surrounding the sample locations. Photographs also will provide a record of the spatial relationships between the sampled area and surrounding features and structures.

4.7.3 Sample Data Sheets

A field sampling form will be completed for each sample location (Appendix C). The form will record the sample ID and time of sample collection.

4.8 Cultural Resource Monitoring

An archaeologist will observe the soil sampling process throughout collection of the surface soil samples. The archaeologist will monitor the sample collection process in an effort to protect cultural resources from disturbance. Pre-sampling records search, site reconnaissance, and the monitoring and reporting of any artifacts, if found, is described in the cultural resources monitoring and reporting protocol provided in Appendix D.

The methodologies described in the cultural resources monitoring and reporting protocol are consistent with standard cultural resources monitoring practices and the LEKT Monitoring and Discovery Plan, which has been provided to E & E by the LEKT. Cultural resources monitoring procedures were approved during meetings between E & E archaeologist Sandra Pentney, LEKT archaeologist Bill White, and City of Port Angeles archaeologist Derek Beery on May 1, 2008.

One archaeologist will be present with each sampling team. The archaeologist will each have stop work authority should any cultural resources be encountered during the sampling. All sample locations will be examined prior to any ground cover removal and the cleared surface will also be examined prior to the sampling being conducted although the archaeologist will not touch the area to be sampled.

In the event that artifacts are found, the find will be documented and properly recorded on Washington State forms, and the artifact will be returned to the hole from which it was recovered. The LEKT archaeologist, City archaeologist, and land owner will be notified within 24 hours of the find. Should a find be recovered in any subsample, the sample will not be collected at that location; a new location will be selected.

Since all artifacts encountered are the property of the landowner from which the artifact is recovered, the landowner will be offered the artifacts. The land owner, LEKT, City, and Ecology will be provided with copies of all of the documentation associated with the find, including the isolate record form required by the DAHP within 14 days of the find.

In the event that human remains are encountered in any of the samples, the E & E archaeologist will immediately notify the City Police, the City archaeologist, and the Clallam County Coroner. The LEKT and the DAHP will also be notified should it be determined that the remains are of Native American origin. Documentation (photographs) of human remains will not be collected until approval is issued by the City archaeologist and/or the LEKT.

4.9 Disposal of Investigation-Derived Waste

4.9.1 Disposal of Incidental Trash

Incidental trash generated during this investigation (including discarded nitrile gloves, aluminum foil, used bowls and spoons, and paper towels) will be placed in plastic trash bags and disposed of as solid waste.

4.9.2 Decontamination Water Disposal

Soap and water decontamination solutions will be poured onto the ground or into a sanitary sewer system at the end of each day.

5.0 Sample Handling Procedures

This section discusses the sample handling procedures, including sample identification, documentation, custody procedures, and sample packing and shipping.

5.1 Sample Identification and Documentation

This section describes procedures for sample identification and chain of custody that will be used for the field activities. The purpose of these procedures is to ensure that the quality of samples is maintained during collection, transportation, storage, and analysis. All chain-of-custody requirements comply with E & E's standard operating procedures (SOPs) for sample handling (Appendix E).

Sample documentation for custody purposes includes:

- Sample identification numbers,
- Sample labels,
- Custody seals,
- Chain-of-custody records,
- Field logbooks, and
- Analytical records.

During the field effort, the site manager or delegate is responsible for maintaining an inventory of these sample documents. This inventory will take the form of a cross-referenced matrix of the following:

- Sample location,
- Sample identification number,
- Analyses requested and request form number(s),
- Chain-of-custody record number, and
- Air bill numbers.

Brief descriptions of the major sample identification and documentation records and forms are provided below.

5.1.1 Sample Identification

Each sample will be assigned a unique alphanumeric identifier describing the sample location. The sample identifier will be recorded on a sample label, which will be affixed to the sample jar, and it will consist of seven digits, representing coded information presented below.

Digits	Description	Code
1,2	Sample Area	W1, W2, W3, E1, E2, E3, E4 (zone) T1, T2, T3 (upslope transect) FF (focused forested area) RD (urban roadside area)
3,4	Consecutive Sample Number	01 (first sample within the sample area) For grid areas, this number also will represent the grid cell number.
5,6	Matrix code	SS (surface soil) RP (field replicate)

The sample collected from zone W1 cell “05” would be labeled as “W105SS” while the second sample collected from transect area two would be labeled as “T202SS.” A field replicate of this sample would be labeled as “T202RP.”

5.1.2 Sample Labels

Sample labels attached to or fixed around the sample container will be used to identify all samples collected in the field. Sample information will be printed legibly. Field identification will be sufficient to enable cross-reference with the sample forms. For chain-of-custody purposes, all QA/QC samples will be subject to exactly the same custodial procedures and documentation as investigative samples.

Each sample label will be written in waterproof ink, attached firmly to the sample containers, and protected with clear tape. The sample label will contain the following information:

- Project name,
- Sample identifier,
- Date and time of collection, and
- Analyses required.

5.1.3 Custody Seals

Custody seals are preprinted, adhesive-back seals with security slots designed to break if the seals are disturbed. Sample shipping containers (e.g., coolers, drums, and cardboard boxes, as appropriate) will be sealed in as many places as necessary to ensure security. Seals will be signed and dated before use. Upon their arrival at the laboratory, the custodian will check (and certify by completing the package receipt log) that seals on shipping containers are intact.

5.1.4 Chain-of-Custody Records

The chain-of-custody records will be completed fully at least in duplicate by the field technician designated by the site manager as responsible for sample shipment to the appropriate laboratory.

Information specified on the chain-of-custody record will contain the same level of detail found in the site logbook, except that the on-site measurement data will not be recorded. The custody record will include, among other things, the following information:

- Name and company or organization of person collecting the samples,
- Date of sample collected,
- Type of sampling conducted (composite/grab),
- Location of sampling station (using the sample code system described in Section 5.1.1),
- Number and type of containers shipped,
- Analysis requested, and
- Signature of the person relinquishing samples to the transporter, with the date and time of transfer noted, and signature of the designated sample custodian at the receiving facility.

If samples require rapid laboratory turnaround, the person completing the chain-of-custody record will note these or similar requirements in the remarks section of the custody record.

The relinquishing individual will record all shipping data (e.g., air-bill number, organization, time, and date) on the original custody record, which will be transported with the samples to the laboratory and retained in the laboratory's file. Original and duplicate custody records with the air bill or delivery note constitute a complete custody record. It is the site manager's responsibility to ensure that all records are consistent and that they are made part of the permanent job file.

5.1.5 Field Logbooks and Data Forms

Field logbooks (or daily logs) and field data forms are necessary to document daily activities and observations. Documentation will be sufficient to enable participants to reconstruct events that occurred during the project accurately and objectively at a later time. All daily logs will be kept in a bound notebook containing numbered pages, and all entries will be made in waterproof ink, dated, and signed. No pages will be removed for any reason.

Minimum logbook content requirements are described in E & E's SOP titled, Preparation of Field Activities Logbooks (see Appendix E). If corrections are necessary, they will be made by drawing a single line through the original entry (so that the original entry is still legible) and writing the corrected entry alongside it. The correction will be initialed and dated. Corrected errors may require a footnote explaining the correction.

5.1.6 Photographs

Photographs will be taken as directed by the team leader. Documentation of a photograph is crucial to ensure its validity as a representation of an existing situation. The following information concerning photographs will be noted in the field logbook:

- Date, time, and location photograph was taken,
- Weather conditions,
- Description of photograph,
- Reasons photograph was taken,
- Sequential number of the photograph, and
- Direction.

After the photos are processed, the information recorded in the field logbook will be summarized in captions in the digital photo log.

5.2 Custody Procedures

The primary objective of chain-of-custody procedures is to provide an accurate written or computerized record that can be used to trace the possession and handling of a sample from collection to completion of all required analyses. A sample is considered to be in custody if it is:

- In someone's physical possession,
- In someone's view,
- Locked up, or
- Kept in a secured area that is restricted to authorized personnel.

5.2.1 Field Custody Procedures

The following guidance will be used to ensure proper control of samples during fieldwork:

- As few people as possible will handle samples;
- Coolers or boxes containing cleaned bottles will be sealed with a custody tape seal during transport to the field or while in storage before use. Sample bottles from unsealed coolers or boxes, or bottles that appear to have been tampered with, will not be used;
- The sample collector will be responsible for the care and custody of samples until they are transferred to another person or dispatched properly under chain-of-custody rules;
- The sample collector will record sample data in the field logbook; and
- The site team leader will determine whether proper custody procedures were followed during the fieldwork and decide whether additional samples are required.

When transferring custody (i.e., releasing samples to a shipping agent), the following will apply:

- The coolers in which the samples are packed will be sealed and accompanied by chain-of-custody records. When transferring samples, the individuals relinquishing and receiving them must sign, date, and note the time on the chain-of-custody record. This record documents sample custody transfer;
- Samples will be dispatched to the laboratory for analysis with separate chain-of-custody records accompanying each shipment. Shipping containers will be sealed with custody seals for shipment to the laboratory. The method of shipment, name of courier, and other pertinent information will be entered in the chain-of-custody record;
- All shipments will be accompanied by chain-of-custody records identifying their contents. The original record will accompany the shipment. Carbon copies will be provided to the project manager; and
- If sent by common carrier, a bill of lading will be used. Freight bills and bills of lading will be retained as part of the permanent documentation.

5.2.2 Laboratory Custody Procedures

A designated sample custodian at the laboratory will accept custody of the shipped samples from the carrier and enter preliminary information about the package into a package or sample receipt log, including the initials of the person delivering the package and the status of the custody seals on the coolers (i.e., broken versus unbroken).

5.3 Sample Handling, Packaging, and Shipping

The transportation and handling of samples must be accomplished in a manner that not only protects their integrity but also prevents any detrimental unnecessary exposure to sample handlers due to the possibly hazardous nature of the samples. Regulations for packaging, marking, labeling, and shipping hazardous materials are promulgated by the United States Department of Transportation (DOT) in 49 Code of Federal Regulations 171 through 177 and/or the International Air Transport Association (IATA) Regulations for Dangerous Goods.

5.3.1 Sample Packaging

Samples must be packaged carefully to avoid breakage or contamination and must be shipped to the laboratory at proper temperatures. The following sample package requirements will be followed:

- Sample container lids must never be mixed. All sample lids must stay with the original containers;
- All sample containers will be placed in a plastic bag to minimize leakage in case a container breaks during shipment;
- The samples will be cooled by placing ice in sealed plastic bags. Ice is not to be used as a substitute for packing materials;

- Any remaining space in the sample shipping container should be filled with inert packing material. Under no circumstances should material such as sawdust, newspaper, or sand be used; and
- The custody record must be sealed in a plastic bag and placed in the shipping container. Custody seals must be affixed to the sample cooler.

5.3.2 Shipping and Containers

The appropriate shipping container will be determined by DOT or IATA regulations for the anticipated level of suspected contaminants. Shipping containers are to be custody-sealed for shipment as appropriate. The custody seals will be affixed in such a way that access to the container can be gained only by breaking a seal.

Field personnel will make arrangements for transportation of samples to and receipt by the laboratory. When custody is relinquished to a shipper, field personnel will telephone the laboratory sample custodian to inform him or her of the expected time of arrival of the sample shipment and to advise him or her of any time constraints on sample analysis.

Samples shipped to Axys will be sent via FedEx, which provides the most efficient cross-border shipping services. Samples shipped to ARI will be sent via a courier or FedEx.

5.3.3 Marking and Labeling

Suggested guidelines for marking and labeling shipping containers are presented below. In all cases, DOT or IATA regulations should be consulted for appropriate marking and labeling requirements, which include the following:

- Use abbreviations only where specified; and
- After a shipping container is sealed, two chain-of-custody seals will be placed on the sample container lids, one on the front and one on the back. To protect the seals from accidental damage, clear strapping tape will be placed over them.

6.0 Laboratory Analytical Methods

Analysis of all soil samples will include dioxin/furan congeners and TOC. Electronic results will be delivered to E & E upon project completion. Table 6-1 summarizes laboratory instrumentation and methods to be used for the soil sample analyses. Table 6-2 provides the analytical methods, preservation, holding time, and container type for each analysis.

The respective laboratory analysts will be responsible for ensuring that appropriate sample analysis procedures are followed and for taking appropriate actions to ensure deficiency correction.

6.1 Dioxin/Furan Analyses

Dioxin/furan analyses will include quantification for the ten homologue groups and 17 congeners with chlorine substitution in the 2, 3, 7, and 8 positions using EPA Method 1613B. Analysis turn-around time will be approximately 45 days. Axys Analytical Laboratory will provide modified reporting limits that are lower than the minimum level defined by EPA Method 1613B, shown in Table 6-3. Dioxin/furan analyses will be performed on all investigative samples, 100 of which will be collected. In addition, five replicate samples will be collected at a frequency of one per 20 samples resulting in a total of 105 dioxin/furan analyses.

6.2 Total Organic Carbon

Organic carbon in soils comes from decaying natural organic matter (humic acid, fulvic acid, amines, urea, and so forth) as well as from synthetic sources such as detergents, fertilizers, and pesticides. TOC is a potentially useful measure in soil because dioxins/furans adsorb to organic matter in soils. Thus, the greater the TOC content in soil, the greater the capacity of the soil to retain dioxins/furans. TOC will be analyzed in all soil samples by ARI by EPA Method 9060. The turn-around time will be approximately 30 days. TOC analyses will be performed on all investigative samples, 100 of which will be collected. In addition, five replicate samples will be collected at a frequency of one per 20 samples resulting in a total of 105 TOC analyses.

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7.0 Quality Assurance Procedures

The purpose of the project QA/QC is to provide confidence in project data results through a system of quality control performance checks with respect to data collection methods, laboratory analysis, data reporting, and appropriate corrective actions based on established performance and data quality criteria. This section presents QA/QC procedures to ensure that the investigation data results are defensible and usable for their intended purpose.

Data quality objectives (DQOs) guide decisions and processes for the collection, analysis, and evaluation of data in order to satisfy overall project objectives. The objectives for the project are to determine the magnitude of dioxin/furans contamination in surface soils and to determine the relative contribution to measured soil dioxin/furans concentrations from former Rayonier Mill emissions compared to other potential sources.

7.1 Data Use

PCDD/PCDF and total organic carbon will be analyzed by Axys Analytical Services and ARI, respectively. Analytical methods and sample numbers are provided in Table 6-1.

The method for dioxins/furans was selected to provide low enough detection limits to evaluate the results with respect to source(s), as well as enable reliable comparisons to existing data. TOC data will be used to normalize dioxin/furans results to organic carbon. Dioxin/furan compounds and method detection limits for this project are provided in Table 6-3.

Data generated by the laboratory will be used for a chemometric data evaluation to investigate source contributions to soil dioxin/furans concentrations. In addition, an assessment of the correlation between TOC and dioxin/furans concentration will be completed.

7.2 Measurement Quality Objectives

Measurement objectives for data quality are presented in Tables 7-1, 7-2, and 7-3, which outline the minimum requirements for data quality. The information provided in Table 7-1 includes project-specific objectives for measurement data by analytical parameter, including laboratory practical reporting limits, and quality control limits established for sample replicates (i.e., laboratory duplicates and triplicates), laboratory matrix spike (TOC only), labeled compound recovery (dioxin/furan only), and control standard analyses.

The following sections describe the methods used to assess data quality.

7.2.1 Precision

Precision is defined as the reproducibility of measurements under a given set of conditions. It is a quantitative measure of variability of a group of measurements with respect to a mean value. Based on the QC samples (i.e., lab duplicates), a measure of bias within the system can be estimated.

Precision is independent of the error (accuracy) of the analyses, and reflects only the degree to which the measurements agree with one another, not the degree to which they agree with the "true" value for the parameter measured. Precision of laboratory duplicates is calculated in terms of relative percent difference (RPD), which is expressed as follows:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2) / 2}$$

where: RPD = relative percent difference
C₁ = larger of two values
C₂ = smaller of two values.

Precision of laboratory triplicates is calculated in terms of relative standard deviation (RSD), which is expressed as follows:

$$RSD = \frac{\sqrt{\frac{1}{n-1} \left[\sum_{i=1}^n (x_i - \bar{x})^2 \right]} \times 100\%}{\bar{x}}$$

where: RSD = relative standard deviation
n = number of measurements
x_i = sample measurement
 \bar{x} = arithmetic mean of x_i measurements

RPD and RSD values must be compared to the criteria established in Table 7-1. For concentrations less than 5 times the detection limit, RPD and RSD criteria are not valid; variations may be as great as 2 times the reporting limit.

When laboratory duplicate RPD values exceed established control limits, the analyst or his/her supervisor must investigate why the data exceed stated acceptance limits and report these findings to the laboratory QA/QC Coordinator. RPD values outside the established control limits may indicate some assignable cause other than normal measurement errors, and the need for corrective action. Follow-up action can include recalibration, reanalysis of the original or duplicate sample, or flagging the data as suspect if problems cannot be resolved.

7.2.2 Accuracy

Accuracy is the degree of agreement of a measurement or average of measurements with an accepted reference or "true value," and is another measure of bias in the system. Accuracy will be assessed based on analyses of method blanks, matrix spikes (TOC only), labeled compound recovery (dioxin/furan only), and control standards. The values for method blanks will not exceed the reporting limit. The percent recovery of matrix spikes will meet the criteria specified in Table 7-1. Percent recovery for matrix spikes and labeled compounds will be calculated using the following equation:

$$\%R = \frac{(S - U) \times 100\%}{C_{sa}}$$

where: %R = percent recovery
 S = measured concentration in spike sample
 U = measured concentration in unspiked sample
 C_{sa} = actual concentration of spike added.

If the analyte is not detected in the unspiked sample, then a value of zero will be used in the equation.

Percent recovery for control standards will be calculated using the following equation:

$$\%R = M/T \times 100$$

where: %R = percent recovery
 M = measured value
 T = true value.

Acceptance criteria, also termed control limits, are based on previously established (i.e., historical) laboratory capabilities for similar samples using control chart techniques. In this approach, the control limits reflect the minimum and maximum recoveries expected for individual measurements for an in-control system. Recoveries outside the established control limits indicate some assignable cause, other than normal measurement error, and the possible need for corrective action. Corrective action could include recalibration of the instrument, reanalysis of the QC sample, reanalysis of the samples in the batch, or flagging the data as suspect if the problem cannot be resolved. These results will be provided in the project report.

7.2.3 Representativeness

Representativeness describes how well the data reflect site conditions in the vicinity of the data point at the time of collection. Representativeness may be maintained or attained by careful documentation of data collection procedures and adherence to standard data collection protocols.

The characteristics of representativeness are usually not quantifiable. Subjective factors to be taken into account are as follows:

- Homogeneity of the site being monitored,
- Homogeneity of a sample taken from one point in the site being monitored, and
- Available information on which a sample plan is based.

The design of the sampling program is to ensure sample locations are selected properly, sufficient numbers of samples are collected to accurately reflect conditions at the site, and samples are representative of the sampling locations. When applicable, a sufficient volume of

sample will be collected at each sampling station to minimize bias or errors associated with sample particle size and heterogeneity.

7.2.4 Completeness

The target value for completeness of all parameters is 95 percent. Measurement data completeness is a measure of the extent that the database resulting from a sampling and analysis effort fulfills the objectives for the amount of data required. For this program, completeness will be defined as the valid data percentage of the total tests requested as follows:

$$\text{Completeness(\%)} = \frac{\text{No. of Successful analyses} \times 100\%}{\text{No. of Requested Analyses}}$$

Successful analyses are defined as those in which the sample arrived at the laboratory intact, properly preserved, in sufficient quantity to perform the requested analyses, and accompanied by a completed Chain-of-Custody form. Furthermore, the sample must be analyzed within the specified holding time and not be rejected according to QC acceptance criteria.

Completeness for the entire project also involves elements specific to field and laboratory documentation of sample collection. This includes documentation detailing whether samples and analyses have been processed using the procedures outlined in this QAPP and whether laboratory SOPs have been implemented.

7.2.5 Comparability

Comparability is the degree to which data from separate data sets may be compared. For instance, sample data may be compared to data from background locations, to established criteria or guidance, or to data from earlier sampling events. Comparability is attained by careful adherence to standardized sampling and analytical procedures, based on rigorous documentation of sample locations (including depth, time, and date).

The use of standardized methods to collect and analyze samples, along with instruments calibrated against National Institute for Standards and Technology (NIST) and U.S. EPA-traceable standards will also ensure comparability, particularly for comparison of data collected from this study (within-study comparability). As discussed in the SSP (E & E 2008), between-study comparability may be limited due to the varying sample collection and analysis methods used for studies in Port Angeles. Differences in the methods selected for this study compared to other Port Angeles studies are based on differences in study objectives.

Comparability also depends on other data quality characteristics. Only when data are judged to be representative of the environmental conditions, and when precision and accuracy are known, can data sets be compared with confidence.

7.3 Quality Assurance and Quality Control for Soil Samples

Laboratory QC samples, used to evaluate the data precision, accuracy, representativeness, completeness, and comparability of the analytical results, are discussed in the following sections.

Analytical performance is monitored through QC samples and spikes, such as laboratory method blanks, labeled compound spikes, QC check samples, matrix spikes, and laboratory duplicate samples. All QC samples are applied on the basis of a laboratory batch. Two basic types of batches are used: the preparation batch and the run (i.e., analytical) batch. The preparation batch includes all samples processed as a unit during sample preparation. Preparation batches do not exceed 20 samples excluding associated QC samples. The QC samples associated with sample preparation include method blanks, laboratory control samples (LCS), matrix spikes, and duplicates. The run batch includes all samples analyzed together in the run sequence. The run sequence is typically defined by the analytical method. For TOC analysis, the run batch is equivalent to the preparation batch. The QC samples associated with the run sequence include calibration standards, instrument blanks, and reference standards.

Instances may arise where high sample concentrations, nonhomogeneity of samples, or matrix interferences preclude achieving the detection limits or associated QC target criteria. In such instances, data will not be rejected a priori, but will be examined on a case-by-case basis. The laboratory will report the reason for deviations from these detection limits or noncompliance with QC criteria in the case narrative.

7.3.1 Laboratory Method Blanks

A laboratory method blank is an analyte-free material processed in the same manner and at the same time as a project sample. The laboratory method blanks serve to demonstrate a contamination-free environment in the laboratory. The goal is for method blanks to be free of contamination. Low level contamination may be present, but must be less than the PQL as defined by the method SOP. If contamination is greater, the samples are reanalyzed. If contaminants are present in the method blank but not in project samples, no further action is required. All sources of contamination that are not common laboratory contaminants as defined in the method SOPs must be investigated as part of the corrective action process. Sample results must not be blank-subtracted unless specifically required by the analytical method.

7.3.2 Labeled Compound Standards

For dioxin/furans, all samples, including the laboratory method blank and standards, are spiked with a set of specific labeled compound standards to monitor the accuracy of the analytical determination. Labeled compound spikes are added at the start of the laboratory preparation process.

Labeled compound recoveries must be within QC criteria for method blanks and LCSs to demonstrate acceptable method performance. If recoveries are outside QC criteria for method blanks or LCSs, corrective action is required and the QC Manager should be notified. Labeled compound recoveries in the samples indicate the method performance on the particular sample matrix. Surrogate recoveries that are outside QC criteria for a sample indicate a potential matrix effect. Matrix effects must be verified based on review of recoveries from the method blank or LCS, sample reanalysis, or evaluation of interfering compounds.

7.3.3 Laboratory Control Sample

An LCS consists of a method blank spiked with target compounds of interest near the mid-point of the calibration range. The LCS is processed by the same sample preparation, standard addition, and analysis as the project samples. The recovery of target analytes in the LCS is an estimation of method accuracy.

LCS recovery must be within the control limits to demonstrate acceptable method performance. If the LCS recovery values are outside QC criteria for the target analytes, recovery values are significantly low, or the compounds were detected in the samples, then corrective action is required. After corrective action is complete, sample re-analysis is required for the failed parameters. For any deviations from the LCS control limits that cannot be resolved by sample re-analysis within holding times, the QC Manager must be notified immediately. If critical samples are affected, the Project Manager may determine that re-sampling is required.

7.3.4 Matrix Spike Sample

A matrix spike (MS) sample consists of a project sample split into two parts and processed as two separate samples in a manner identical to that of the rest of the samples. In addition to the regular addition of monitoring standards (internal standards, surrogate), spiking analytes are added to the sample aliquot. An MS must be prepared for every batch of 20 samples (or fewer) for a given matrix if sufficient sample allows. The laboratory must analyze a site-specific MS samples for every batch that contains samples from the site, even if the batch contains samples from other sites.

MS recovery values are a measure of the performance of the method on the sample being analyzed. MS recovery values outside the control limits indicate matrix effects. The laboratory should notify the QC Manager of these instances to determine an appropriate corrective action.

7.3.5 Duplicate Sample

A duplicate sample consists of a set of two samples obtained in an identical manner from the same project sample. The collection of duplicate samples from a heterogeneous matrix requires homogenization to ensure that representative portions are analyzed. One sample per batch of 20 samples or fewer per matrix is analyzed.

The duplicate is prepared for methods that typically show concentrations of target analytes above MDLs, such as TOC. The RPD values between the recovery values in the original and duplicate measure the precision of the analytical method on the actual project samples. For this project, QC criteria for RPDs are 20 percent.

7.3.6 Performance Evaluation Samples

As part of the laboratory approval process, the laboratory must analyze external performance evaluation (PE) samples provided by an outside certifying agency on an annual basis. The laboratory must maintain acceptable scores on PE samples as part of the approval process. For

this project any PE failures for project target compounds must be reported to the QC Manager immediately.

7.4 Data Validation

Axys and ARI will provide data supported by sufficient backup and QA information to permit an independent determination of data quality by EcoChem, Inc. Deliverables submitted by the laboratory will include the information described below.

- A case narrative that includes a summary of any quality control, sample, shipment, or analytical problems, as well as documentation of all internal decisions. Problems will be outlined and final solutions documented. A copy of the signed Chain-of-Custody form for each group of samples will be included in the narrative packet.
- Sample concentrations reported on standard data sheets in proper units and to the appropriate number of significant figures (i.e., two significant figures for concentrations less than 10 and three significant figures for concentrations greater than 10). For undetected values, the lower limit of detection of each compound will be reported separately for each sample. Date of sample analysis must be included.
- Laboratory duplicate results (TOC only)
- Matrix spike results (TOC only)
- Labeled compound recovery results (dioxin/furans only)
- Control standard results
- A method blank summary.

Data will be compared to the project data quality objectives (refer to Tables 7-1 through 7-3) to determine if the data are sufficient to meet specified project objectives.

The analytical laboratory will demonstrate its ability to produce acceptable results using the recommended methods. Data will be evaluated based on the following criteria:

- Performance on method tests
- Adequacy of detection limits obtained
- Precision of duplicate analyses
- Comparison of the percentage of missing or undetected substances among duplicate samples
- Percent recovery of spike compounds.

Routine procedures for measuring precision and accuracy include use of duplicate analyses, control standards, matrix spikes, and procedural blanks. Duplicates, matrix spikes, and method blanks will be analyzed routinely by the laboratory.

After receipt from the laboratory, project data will be validated by EcoChem, Inc. as described in the following section.

7.4.1 Evaluation of Completeness

The QC Manager verifies that the laboratory information matches the field information and that the following items are included in the data package:

- Chain-of-custody forms,
- Case narrative describing any out-of-control events and summarizing analytical procedures,
- Data report forms,
- QA/QC summary forms,
- Calibration summary forms, and
- Chromatograms documenting any QC problems.

If the data package is incomplete, the QC Manager contacts the laboratory, which must provide all missing information within one day.

7.4.2 Evaluation of Compliance

The actual data validation follows the procedures that are briefly outlined below:

- Review the data to check field and laboratory QC results to verify that holding times and acceptance and performance criteria were met, and to note any anomalous values;
- Review chromatograms, mass spectra, and other raw data if provided as backup information for any apparent QC anomalies;
- Ensure all analytical problems and corrections are reported in the case narrative and that appropriate laboratory qualifiers are added;
- For any problems identified, review concerns with the laboratory, obtain additional information if necessary, and check all related data to determine the extent of the error; and
- Apply data qualifiers to the analytical results to indicate potential limitations on data usability.

QC Managers will follow qualification guidelines in applicable USEPA National and Regional Data Review guidelines (USEPA 2005, 1996, 1994).

7.4.3 Data Validation Reporting

The QC Manager will perform the following reporting functions:

- Alert the Project Manager to any QC problems, obvious anomalous values, or discrepancies between the field and laboratory data, and resolve any issues;
- Discuss QC problems in a data validation memo for each laboratory report, the data validation memo and copy of the data package will be sent to the Project Manager;
- Review the laboratory electronic data deliverable (EDD) and electronic field data, enter the data qualifiers into the database, and prepare analytical data summary tables. (The tables will summarize those samples and analytes for which detectable concentrations were exhibited as well as complete analytical summary tables – the tables will include field QC samples);
- At the completion of all field and laboratory efforts for site, the QC Manager will prepare a data review/validation memorandum that will summarize planned versus actual field and laboratory activities and data usability concerns.

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8.0 Data Analysis and Reporting

This section describes data analysis and reporting requirements for the data collection activities described above.

8.1 Technical Memorandum

Once data validation is completed, a technical memorandum will be prepared compiling all the results and providing descriptive and exploratory data evaluations. This memorandum will provide a point of departure for more detailed chemometric data evaluations to investigate source contributions to soil dioxin/furan concentrations throughout the study area. The technical memorandum will focus on the sampling results, introduced only by a brief summary of the study purpose and design.

The technical memorandum will provide complete data tables with relevant data qualifiers assigned by the validation subcontractor. The data set will be graphically summarized using approaches such as concentration histograms, cumulative frequency plots, and/or probability plots. The magnitude and overall spatial pattern of dioxin/furan contamination will be illustrated by mapping the results using color coding for each sampling location to show the range in chemical concentration. In addition, scatter plots will be developed to demonstrate the relationship(s) among distance, direction, and concentration, including overall trends and local variability in concentration. These plots will facilitate review of the data with respect to magnitude and spatial pattern.

Other evaluations will include assessments of the correlation between TOC and dioxin/furan concentration, comparisons of results for residential (disturbed) and wooded (undisturbed) land use types, and preliminary analyses of variability in dioxin/furan profiles using normalized multi-congener values and plots.

After completion of the technical memorandum and submittal to Ecology for review, an appropriate strategy for chemometric evaluation of the data will be assessed and discussed with Ecology. During this period, Ecology will send individual dioxin/furan results to study participants along with a letter describing the significance of the results. Data also will be uploaded to Ecology's EIM system.

The following will be completed as part of the technical memorandum:

- GIS map showing toxicity equivalency concentration (TEQs) and/or total dioxin/furans for collected samples
- Summary statistics (geometric mean, arithmetic mean, standard deviation, variance, etc.)
- Descriptive statistics (homogeneity, cumulative frequency plots, probability plots, histograms, and box-whisker plots by congener and homologue group)
- Distribution testing

- Normalization calculations (normalize congener concentration by TOC, homologue, total dioxin/furans concentration)
- Comparison and correlation of developed versus undeveloped properties using probability plots, box-whisker plots, histograms
- Correlation between TOC and concentration as TEQ
- TEQ calculations (various treatments for nondetected values)
- Plot concentration by distance from stack
- Scatter plots of congener versus congener concentrations (all combinations)
- Scatter plots of distribution versus concentration as TEQ
- Scatter plots of direction, distance versus concentration as TEQ

8.1.1 Application of Toxic Equivalency Factors and Calculation of TEQs

Chemical concentrations for dioxins/furans are usually modified by a toxic equivalency factor (TEF) and then each class of chemical is summed to obtain a TEQ. The TEF is an estimate of the relative toxicity of a specific 2,3,7,8-substituted congener compared to the reference chemical, 2,3,7,8-TCDD. The TEQ represents the total contribution of the individual congeners for total dioxin/furan toxicity. The TEFs, listed in Table 708-1 of the MTCA Cleanup Regulation (WAC 173-340-900), developed by the World Health Organization (Van den Berg et al. 2006) will be used to calculate the TEQ. A list of TEFs and a more complete discussion of this methodology is provided in *Evaluating the Toxicity and Assessing the Carcinogenic Risk of Environmental Mixtures Using Toxicity Equivalency Factors* (Ecology 2007b).

8.1.2 Treatment of Nondetect Results for Calculation of TEQs

A U qualifier indicates that the analyte was not detected above the method detection limit (MDL). The MDL is the lowest concentration that can be reliably measured and reported with 99 percent confidence that the value is greater than zero (Ecology 2007a).

Three approaches will be followed to describe concentrations of dioxins/furans. One approach will involve using one-half of the MDL for nondetected congeners when calculating the total toxicity equivalent concentration (TEQ) for each sample. The second approach will involve assuming a value of zero for nondetected congeners when calculating the TEQ. The third approach includes calculation of the TEQ using the full MDL concentration for nondetected congeners. Results using all approaches will be presented in the technical memorandum.

8.2 Final Project Report

A project report will be developed documenting the study objectives, protocols, and results. That report will include validated data and calculated TEQ values, summary and descriptive statistics and data visualizations, georeferenced concentration maps, any deviations from the SSP and SQAP, and a discussion of the chemometric data evaluation process, including source identification analysis results.

Details of the chemometric evaluations will be provided as an appendix to the final project report. As noted earlier, resident names and addresses will not be included in the final project report; however, sample locations will be presented on maps. The public review draft report will be submitted to Ecology and then finalized following a public comment period.

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