APPENDIX A

SAMPLING AND ANALYSIS PLAN FOR HUMAN HEALTH AND ECOLOGICAL RISK ASSESSMENT

ST. REGIS PAPER COMPANY SITE
CASS LAKE, MINNESOTA

July 29, 2004

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1. INTRODUCTION

This document presents the sampling and analysis plan (SAP) for a field investigation scheduled for summer of 2004 to support the human health and ecological risk assessments of the St. Regis Paper Company Site (the Site), which includes the Cass Lake City Dump area in Cass Lake, Minnesota (Figure 1). This SAP is not intended to support further characterization of the site for removal purposes. If the risk assessment indicates additional site characterization for a removal action is necessary, an SAP specific to the removal action will be prepared. The St. Regis Paper Company Site is a closed wood-treating facility that is listed on the National Priorities List (NPL) of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), which is administered by the U.S. Environmental Protection Agency (EPA). The City Dump Pit area is located at a former city dump owned and operated by the City of Cass Lake, south of the St. Regis Paper Company Site. The Site is currently in the operation, maintenance, and monitoring stages following the implementation of remedial actions in the mid-1980s. EPA Region 5 assumed regulatory authority over the Site from the Minnesota Pollution Control Agency in 1995. In September 2000, EPA completed a 5-Year Review of the Site and concluded that additional investigation was needed to address identified data gaps.

International Paper Company (International Paper) is cooperating with EPA Region 5 by voluntarily developing human health and ecological risk assessments for the Site. The risk assessments will provide a more complete understanding of the nature and magnitude of risks to human health and the environment at the Site, as well as support decision-making regarding the protectiveness of the existing remedial actions and the need, if any, for additional action. Detailed discussions of Site history and risk assessment approach are presented in the Risk Assessment Work Plan (RAWP).

1.1 TECHNICAL APPROACH AND RATIONALE

The objective of this investigation is to collect data to support the human health and ecological risk assessments, as planned in the RAWP. The RAWP presents detailed conceptual site models (CSMs) that describe chemical sources, release mechanisms, transport pathways, and potential routes of exposure for human and ecological receptors on and in the vicinity of the Site. The RAWP also identifies the list of human health chemicals of potential concern (COPCs) and chemicals of potential ecological concern (COPECs). The lists of COPCs and COPECs presented in the RAWP are considered preliminary and will be updated based on analytical results from the samples proposed for collection in this plan.

From the CSMs, COPCs, and COPECs, EPA's 7-step Data Quality Objectives (DQO) process[1] was followed to focus the sampling effort on data gaps relevant to the risk assessment effort. The DQO process identified data gaps in soil, sediment, surface water, and biota on and in the vicinity of the Site. This SAP and its supporting appendices present the detailed plans for filling these data gaps, including sampling locations, sampling matrices, target analyte lists, sampling methods, and analytical methods.

1.2 ORGANIZATION OF THE DOCUMENT

In addition to this introduction, this SAP consists of the following sections:

- Section 2 – Sampling Activities. The proposed sampling activities are presented by matrix (e.g. soil, sediment, garden produce, etc.) and grouped into terrestrial and aquatic sampling. The discussion associated with each matrix includes a brief description of the sampling objective, followed by an explanation of the technical approach.
- Section 3 – Methods and Quality Assurance. A brief discussion of the sampling methods and quality assurance plan is presented.
- Section 4 – Schedule. The tentative schedule for the sampling effort is presented, noting specific timing constraints.
- Section 5 – References.

A detailed Field Sampling Plan (FSP) is presented in Attachment A.

[1] Step 1: Identify the problem; Step 2: Identify the decisions; Step 3: Identify inputs to the decisions; Step 4: Identify the Study Boundaries; Step 5: Develop a decision rule; Step 6: Specify limits on decision errors; and Step 7: Optimize the design for obtaining data.

2. SAMPLING ACTIVITIES

The following sections present the scope of work for sampling activities to support the human health and ecological risk assessments. The sampling activities are grouped into terrestrial and aquatic activities, and further subdivided by environmental matrix. The scope of work for each matrix includes relevant sampling activities for all areas and objectives related to that matrix. Detailed sampling procedures are described in the FSP (Attachment A). For administrative purposes within the risk assessments, the Site and the surrounding areas were grouped into two areas: Area A[2] - the former wood-treating area and North Storage Area of the St. Regis Paper Company Site and nearby residences; and Area B[3] - the Southwest Area, City Dump, and nearby surface water bodies. These areas are depicted in Figure 2.

Planned terrestrial field activities include sampling of soil, soil invertebrates, terrestrial forage plants, garden produce, and house dust. Planned aquatic field activities include sampling of sediments, benthic invertebrates, wild rice (and other aquatic plants), surface water, and fish tissue. This SAP discusses the objectives and technical approach for each sampling matrix.

2.1 TERRESTRIAL SAMPLING

This section presents the objectives and technical approach for sampling of each terrestrial sampling matrix (soil, soil invertebrates, terrestrial plants, garden produce, and house dust). It should be noted that in addition to evaluating risks and hazards associated with potential exposure to chemical concentrations measured in garden produce samples, the

[2] Area A consists of certain former operating areas of the St. Regis Paper Company facility (north storage area, bridge fabrication area, raw wood shaping area, treating plant area, administrative area, debarking areas, sawmill area, wastewater ponds, spray/landfill area, and teepee burner area); contiguous lands owned by the City of Cass Lake and the Burlington Northern and Santa Fe (BNSF) Railroad; and the residences in the area bordered by the BNSF railroad to the north, Highway 147 to the west, 3rd Street to the south, and the former operating areas and the forested wetland to the east.

[3] Area B consists of the Southwest Area of the former wood-treating facility property (including the containment vault and former wood storage area), the portion of the Chippewa National Forest south of Area A and north of the City Dump Area, the City Dump Area, Fox Creek, the Pike Bay Shoreline, and the Channel Area. Potential migration of site-related chemicals to surface water and sediment in the Channel Area, Pike Bay, and Cass Lake will also be considered in the Area B evaluation.

human health risk assessment (HHRA) will also evaluate risks and hazards associated with potential exposure to chemical concentrations in garden produce estimated using chemical-specific uptake factors identified in the literature and soil exposure point concentrations (EPC). Analyte lists and sampling areas for the terrestrial sampling are summarized in Table 1. Proposed sample counts for the terrestrial sampling are summarized in Table 2. Sampling methods and quality control are discussed briefly in Section 3 and presented in detail in Appendices A and B.

2.1.1 Soil

2.1.1.1 Objectives

Soil sampling will be performed at the Southwest Area, the former City Dump Area, the Chippewa National Forest, the Forested Wetland, South 2nd Street, the Former Operations Area, and in offsite background/reference locations. The soil sampling is designed to address the following objectives:

- Evaluate the concentrations of COPCs and COPECs in previously uncharacterized portions of the Southwest Area, the former City Dump, the Chippewa National Forest, the Forested Wetland, and South 2nd Street. The data will be evaluated and representative EPCs will be determined. (Note: the proposed characterization, as well as existing sampling conducted in other portions of the site, should be evaluated in a risk assessment context -- that is, in terms of potential current and future exposure scenarios. Additional sampling and characterization of COPCs and COPECs in various areas of the site may be required as part of future removal and remedial design efforts);
- Determine concentrations of COPCs in soil samples collocated with samples of native plants gathered by Native American and other human receptors for consumption, medicinal, and other purposes, and concentrations of COPECs in soil samples collocated with samples of soil invertebrates and grasses. Samples of vegetation will be collected in the Southwest Area (grasses only), the City Dump Area (grasses only), the Chippewa National Forest (gathered plants only), the Forested Wetland (gathered plants only), and the Former Operations Area (grasses only). Soil invertebrate samples will be collected in the Southwest Area, the City Dump Area, and the Former Operations Area.
- Characterize the concentrations of metals and chlorinated pesticides in background/reference area soils;

The soil sampling program presented in this SAP is broadly consistent with the technical approach and sampling methodology developed for the 2003 Removal Site Evaluation and Supplemental Assessment at the Former St. Regis Paper Company Site (Integral and

Barr 2003). This will facilitate the use and comparison of data from the two efforts in the risk assessments.

2.1.1.2 Technical Approach

A grid-based sampling approach will be utilized to collect surface and subsurface soil samples in the Southwest Area, and the City Dump Area. Composite soil samples, to be collocated with soil invertebrate and grass samples, will be collected in these areas. Composite soil samples from background/reference areas will be collected to characterize metals and chlorinated pesticides. Composite samples will be collected from the Forested Wetland (one depth interval only [0 to 20 centimenters]) and from South 2nd Street (to evaluate the application of wastewater as a dust suppressant). Additionally, discrete surface soil samples will be collected in the Chippewa National Forest. The specific soil sampling planned for each area is described below.

2.1.1.2.1 Southwest Area

Composite surface soil samples from the 0- to 4-inches bgs (below ground surface) interval will be collected from the 33 grid cells shown on Figure 3. The Resource Conservation and Recovery Act (RCRA) containment vault will not be sampled. Each composite will consist of five discrete subsamples collected from the four corners and the center of each grid cell. After appropriate sample compositing and homogenization has been completed in accordance with the methods specified in the FSP (Attachment A), each sample will be divided into two split samples. One split sample from each location will be field screened for concentrations of polycyclic aromatic hydrocarbons (PAHs) and pentachlorophenol (PCP) using immunoassay methods; and for metals (As, Cr, Cu, Pb, Hg, and Zn) using x-ray fluorescence (XRF) methods. It should be noted that the field screening mirrors the approach followed as part of sampling conducted in 2001 (Tetra Tech 2002). Also, no single element of the field screening approach (for example, screening for PCP using the immunoassay method) is guaranteed to accurately identify the relative concentrations of compounds not being screened (for example dioxins). However, the collective results of the field screening results will provide adequate information for deciding which samples to submit for definitive quantitative analysis. The other split samples will be archived for laboratory analysis pending the field screening results.

Sixteen archived split samples from the 0- to 4-inch bgs interval will be selected for laboratory analysis based on field screening results. Sample selection will be made to reflect the range of chemical concentrations measured in the field screening and to provide representative spatial distribution over the Southwest Area. Samples selected for laboratory analysis will display relatively low, moderate, and high chemical concentrations based on field screening results. In addition, the selection of samples for

definitive analysis will also be based on obtaining a representative coverage of the portion of the Southwest Area being sampled. Samples submitted for laboratory analysis will be analyzed for dioxins/furans, PAHs, PCP, and target analyte list (TAL) metals (Note: for the purpose of the SAP, TAL metals do not include the following essential nutrients – calcium, magnesium, potassium, and sodium). Samples collocated with soil invertebrate and plant tissue samples (see Section 2.1.2 and 2.1.3) will also be analyzed for total organic carbon (TOC), acid-insoluble residue, pH, cation exchange capacity, and bioassay tests.

Composite subsurface soil samples from the 4- to 12-inch interval will also be collected from the 33 locations and field screened as described for the samples from the 0- to 4-inch bgs interval. Eight archived split samples from the 4- to 12-inch bgs interval will be selected for laboratory analysis based on field screening results as described for the surface samples. Samples submitted for laboratory analysis will be analyzed for dioxins/furans, PAHs, PCP, and TAL metals. Eight additional subsurface samples will be selected for laboratory analysis if, based on professional judgment, the results from the initial 8 subsurface samples exhibit a pattern of soil screening level exceedances (see Tables 3 and 4 which present the relevant human health and ecological screening levels). Deeper soil samples will be collected as necessary during a supplemental field sampling effort.

2.1.1.2.2 City Dump

Composite split soil samples from the 0- to 4-inch bgs interval will be collected from the 21 grid cells shown on Figure 3. Each composite will consist of five discrete subsamples collected from the four corners and the center of each grid cell. After appropriate sample compositing and homogenization has been completed in accordance with the methods specified in the FSP (Attachment A), each sample will be divided into two split samples. One split sample from each location will be field screened for PAHs and PCP using immunoassay methods; and for metals (As, Cr, Cu, Pb, Hg, and Zn) using XRF methods.

Ten archived split samples from the 0- to 4-inch bgs interval will be selected for laboratory analysis based on field screening results. Sample selection will be made to reflect the range of chemical concentrations measured in the field screening and to provide representative spatial distribution over the City Dump Area. Samples submitted for laboratory analysis will be analyzed for dioxins/furans, PAHs, PCP, polychlorinated biphenyls (PCBs) (as Aroclors), chlorinated pesticides, and TAL metals. (Note: "dioxin-like" PCB congeners will be determined in up to two of the archived samples. The samples selected for this additional analysis will be the samples that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). Samples collocated with soil invertebrate and plant tissue samples (see Section 2.1.2 and 2.1.3) will also be analyzed for TOC, acid-

insoluble residue, pH, cation exchange capacity, and bioassay tests. The remaining split samples will be archived for laboratory analysis pending the field screening results.

Composite subsurface soil samples from the 4- to 12-inch interval also will be collected and subject to field screening as described for the 0- to 4-inch bgs interval. Five archived split samples from the 4- to 12-inch bgs interval will be selected for laboratory analysis based on field screening results. Samples submitted for laboratory analysis will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides, and TAL metals. (Note: "dioxin-like" PCB congeners will also be determined in one of the archived subsurface samples. The sample selected for this additional analysis will be the sample that exhibits the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). Five additional subsurface samples will be selected for laboratory analysis if, based on professional judgment, the results from the initial 5 subsurface samples exhibit a pattern of soil screening level exceedances (see Tables 3 and 4). These additional samples will be analyzed for the same suite of analytes described above. Deeper soil samples will be collected as necessary during a supplemental field sampling effort.

2.1.1.2.3 Forested Wetland

Collocated surface soil (0 to 20 cm depth) and surface water (described in Section 2.2.4) samples will be collected from four (4) grid cells in the forested wetland, east of the Former Operations Area (see Figure 5). Each soil composite will consist of 5 discrete subsamples collected from the four corners and the center of each grid cell. Surface water samples will be collected from the center of each grid cell (if no surface water is present at the center of a cell, surface water will be collected from one of the cell corners and documented). After appropriate sample compositing and homogenization has been completed in accordance with the FSP (Attachment A), samples will be analyzed for PCBs (as Aroclors; soil only), dioxins/furans (soil only), PAHs, PCP, TAL metals, grain size (soil only), TOC (soil only), chlorinated pesticides (soil only), and total suspended solids (TSS) (surface water only). (Note: "dioxin-like" PCB congeners will also be determined in one of the archived subsurface samples. The sample selected for this additional analysis will be the sample that exhibits the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed.) Wet sieving methods will be used to obtain an estimate of grain size distribution of all soil samples while in the field.

Analytical results will be screened in the risk assessment following the procedures described in the RAWP. If chemicals of potential concern are identified, the need for

additional sampling and/or quantitative evaluation in the risk assessment will be assessed.

2.1.1.2.4 Chippewa National Forest

Discrete split soil samples from the 0- to 4-inch bgs interval will be collected from approximately the center point of each of the 10 grid cells shown on Figure 3. Each discrete sample will be divided into two split samples. One split sample from each location will be field screened for PAHs and PCP using immunoassay methods; and for metals (As, Cr, Cu, Hg, Pb, and Zn) using XRF methods. Also, two discrete split soil samples from the 0- to 4-inch bgs interval will be collected adjacent to the sewer drain located in the Chippewa National Forest. The split samples will be handled as described above.

Five archived split samples from the 0- to 4-inch bgs interval will be selected for laboratory analysis based on field screening results. Sample selection will be made to reflect the range of chemical concentrations measured in the field screening and to provide representative spatial distribution over the Chippewa National Forest. Samples submitted for laboratory analysis will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides, and TAL metals. (Note: one of the archived samples will also be sampled for PCBs as "dioxin-like" congeners. The sample selected for this additional analysis will be the sample that exhibits the highest Aroclor concentrations; if no sample is identified with "elevated" Aroclor concentrations, the congener analysis will not be performed).

Deeper soil samples will be collected as necessary during a supplemental field sampling effort.

2.1.1.2.5 Former Operations Area

Soil sampling locations at the Former Operations Area will be determined in the field, as described in Section 2.1.2.2. Six composite samples (each sample will be a five-point composite sample collected from 0- to 4-inches bgs) will be collected from the Former Operations Area. Samples will be analyzed for dioxins/furans, PAHs, PCP, TAL metals, TOC, and acid-insoluble residue, pH, cation exchange capacity, and bioassay tests.

2.1.1.2.6 Background/Reference Areas

Eight composite samples (each sample will be a five-point composite sample) will be collected from background/reference areas at a depth interval of 0- to 4-inch bgs. Each sample will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides and TAL metals. (Note: "dioxin-like" PCB congeners will be determined in up

to two of the samples. The samples selected for this additional analysis will be the samples that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed).

Composite soil samples from the 4- to 12-inch interval also will be collected at each of the eight locations. Based on the results of the analyses of the surface soil samples, four subsurface samples (representing locations with elevated concentrations of contaminants in surface samples) will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides and TAL metals. (Note: "dioxin-like" PCB congeners will be determined in up to two of the archived samples. The samples selected for this additional analysis will be the samples that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed).

Background/reference sample locations will be selected by qualified technical staff in the field, applying the following criteria:

- Samples will be located in the general vicinity of Cass Lake (i.e., far enough from the St. Regis site to have not likely been impacted by site-related activities, but close enough to be considered representative of site area conditions).
- Samples will be located on the same or similar soil types as those found at the Site.
- Composite subsamples will be collected from a similar grid size.

Background/reference sample locations will be discussed with and approved by EPA.

2.1.1.2.7 South 2nd Street

Soil samples will be collected from South 2nd Street to assess the impact of the alleged historical application of wastewater as a dust suppressant on the street. Four composite samples will be collected from 4- to 12-inches bgs. The samples will be collected along four transects evenly spaced along the street. Each composite sample will consist of four grab samples collected along the respective transect. Each sample will be analyzed for dioxins/furans, PAHs, PCP, and TAL metals.

2.1.2 Soil Invertebrates

This section presents the objectives and technical approach for (1) collection of soil invertebrates to support the ecological exposure assessment for insectivorous mammals and birds, and (2) evaluating the toxicity of soil to invertebrates. These samples will be collocated with forage plant tissue samples and soil samples. The sampling plan for forage plant tissue is presented in Section 2.1.3.

2.1.2.1 Objectives

The bioavailability of chemicals in soil can vary considerably with soil characteristics; therefore, the RAWP proposed collecting collocated soil, soil invertebrate (i.e., earthworms or grubs), and plant tissue samples from the Site. The collocated samples will be used to characterize concentrations of COPECs in soil invertebrates ingested by mammals and birds and to develop site-specific soil-to-biota accumulation factors (BAFs) that will be used to predict chemical concentrations in soil invertebrates for additional locations. Together, the empirical tissue concentrations and predicted tissue concentrations will be evaluated to determine soil invertebrate EPCs for modeling tissue ingestion exposure by insectivores.

The following section presents the technical approaches for collection of soil invertebrates and evaluation of soil toxicity. The collocated sampling of plant tissue (grasses) is presented in Section 2.1.3, while the collocated sampling of soil is presented in Section 2.1.1.

2.1.2.2 Technical Approach

Locations for collecting soil invertebrates will be selected based on an evaluation of available soil chemical data and results of the field reconnaissance survey of soil invertebrate populations. The field reconnaissance survey will be conducted prior to the commencement of sampling to determine qualitatively the density and spatial distribution of soil invertebrates at the site. It should be noted that the reconnaissance survey will be relatively basic and will include photographic and logbook documentation of field conditions (including types and distribution of soil invertebrates; types and distributions of grasses and gathered plants will also be noted to assist in the selection of sample locations for these media) at potential sampling locations. Available information indicates grubs, typically of the May/June beetle, may be available; the soils do not appear to be generally suitable for earthworms. The target sampling plan for soil invertebrates is collection of six (6) samples from the Former Operations Area, two (2) samples from the Southwest Area, and four (4) samples from the City Dump. However, the availability of soil invertebrates may necessitate a different distribution of sample locations.

For the purpose of deriving site-specific BAFs, it is important to collect samples from locations representing the range of COPEC concentrations. Therefore, sample locations should be selected from areas with low and high concentrations of each COPEC. The ranges of detected concentrations of COPECs in soil sample locations at the Former Operations Area are presented in the FSP (Attachment A). Sampling locations in the Southwest Area and City Dump will be based on the results of the field screening. This information will be used to guide sample location selection.

Five-point composite samples of soil invertebrates will be collected from each location and analyzed for dioxins/furans, PCP, PAHs, PCBs (Aroclors in the City Dump only), TAL metals, lipids, acid insoluble residue, and bioassay tests.

2.1.2.3 Contingency Plan

There is concern that biomass of soil invertebrates may not be sufficient for collecting information on COPEC concentrations in tissue. In the event that biomass is insufficient, in-situ bioaccumulation tests or laboratory bioaccumulation tests (using ASTM Method E 1676-97) may be used. Specific test organisms and test methods (including modifications to standard procedures) will be discussed with EPA.

In addition, the findings of the field reconnaissance survey, the results of definitive tissue sampling, and the results of the definitive soil sampling will be evaluated in the ecological risk assessment to determine the significance of the absence or presence of soil invertebrates at the site. Examples of factors that will be evaluated include COPEC concentrations and distributions, physiochemical characteristics, and food availability (inferred from TOC data).

2.1.2.4 Technical Approach for Evaluation of Soil Toxicity

The screening of existing soil data indicates contaminated soils may be toxic to soil invertebrates. Information also indicates that soils at the site may not support earthworms, a common soil toxicity test organism used as a surrogate for soil invertebrates. The specific factors responsible for low numbers of earthworms are not known; however, reduced food quantity and poor soil structure may be two of the main limiting factors. Therefore, to determine the toxicity of soils, a modified earthworm survival and growth test (based on ASTM Method E 1676-97) or an alternative test, such as the International Standards Organization (ISO) collembola reproduction test (ISO Guideline 11267) (ISO 1999), will be performed with the collocated soil samples discussed in Section 2.1.1. The test organism and specific test procedures will be discussed with EPA.

2.1.3 Plant Tissue

2.1.3.1 Objectives

Plant tissue samples will be collected to determine (1) COPEC EPCs for food ingested by herbivorous and omnivorous receptors identified in the RAWP and (2) COPC EPCs for food ingested by human receptors identified in the RAWP.

2.1.3.2 Technical Approach

Plant tissue samples of grasses will be collected from 12 locations distributed over the Former Operations Area, the Southwest Area, and the City Dump Area. It should be noted that ecological receptors are expected to ingest other plants in addition to grasses. However, the uptake of chemicals into grasses is expected to be at least as great as the uptake of chemicals into other plant parts (e.g., fruits [reproductive portions]). Six plant tissue samples will be collected in the Former Operations Area, two (2) samples will be collected in the Southwest Area, and four (4) samples will be collected in the City Dump Area.

Gathered plant tissue samples from an additional eight (8) locations will be collected in portions of Area B (outside of the Southwest Area and the City Dump Area) and the forested wetland. At these locations, samples will be collocated with composite soil and sediment samples. Plant tissue samples from these eight locations will be archived. The decision to analyze some or all of these plant tissue samples will be based on review of the analytical results associated with the primary media in which the plants grow (i.e. soil and sediment) and the analytical results of homegrown produce and wild rice samples. At a minimum, plant tissue samples collected from locations at which either a soil or sediment human health or ecological screening value (see Tables 3 and 4, respectively) is exceeded will be analyzed. Of the previously identified Area B components, soil samples will be collected in the Chippewa National Forest and the Forested Wetland, and sediment samples will be collected elsewhere. The precise location from which samples will be collected and the distribution of plant species and soil versus sediment samples will be determined in the field in consultation with Leech Lake Band members and local biologists.

Five-point composite samples will be collected from each location and analyzed for dioxins/furans, PCP, PAHs, and TAL metals. Sampling methods are detailed in Attachment A.

2.1.4 Garden Produce

2.1.4.1 Objectives

Sampling of home-grown garden produce and reference produce samples from local farm stand will be performed to support the human health risk assessment. Current and future residents who maintain home gardens within Area A (see Figure 2) may ingest COPCs that are taken up by produce. Although this is a potentially complete exposure pathway, the significance of this pathway is unknown as no produce or vegetation has been sampled previously from home gardens within Area A. To evaluate the significance of this pathway, homegrown produce samples will be collected from residences within Area

A, if such gardens currently exist and access is granted. It should be noted that in addition to considering the analytical results for any garden produce samples collected, the HHRA will also evaluate potential exposure to chemical concentrations in garden produce as estimated using chemical-specific uptake factors identified in the literature and soil EPCs.

2.1.4.2 Technical Approach

Produce samples and collocated composite soil samples (0- to 12-inches bgs) will be collected from home gardens within Area A. For the purpose of this sampling effort, only home gardens in which plants are grown in native soils will be considered. Produce grown in raised beds or in gardens in which off-site soil, compost, or other amendments have been brought in to supplement native soil will not be sampled. Home garden locations, sample quantities, types of produce, and volunteer study participants cannot be determined until the growing season has begun. Produce growing in each sampled garden will be recorded, although all produce types will not be sampled. The most prevalent types of protected (edible portions are covered by a protective covering, e.g. peas, corn, and squashs) and unprotected (protective covering is absent (e.g. lettuce) above-ground produce and below-ground produce (e.g., carrots) will be sampled from each garden to assess COPC uptake via particle deposition, vapor adsorption, and root uptake pathways. Prevalence will be determined in the field in consultation with Leech Lake Band representatives. It is likely that the types of produce sampled in each garden will vary. Produce types with higher vegetable fat content (for example, corn [1.1 grams of vegetable fat/serving], soybeans [5.8 grams/serving – green and 15.4 grams/serving – ripe], parsley [1 gram/serving], and beans [>1 gram/serving]) will be preferentially selected over produce types with lower vegetable fat content (for example, lettuce [0.2 to 0.5 gram/serving] and carrots [0.1 gram/serving]). Produce with higher vegetable fat content may be better indicators of dioxin/furan and PCP uptake, as these chemicals would be expected to partition into fats preferentially.

Five gardens will be sampled, if possible. The laboratory will split the samples. One split will be analyzed for dioxins/furans, PAHs, PCPs, and arsenic; the other will be archived for future additional analyses, if necessary. Sample collection and preparation methods and equipment decontamination methods are presented in Attachment A.

Composite garden soil samples from the 0- to 12-inch bgs interval will be collected from each garden where produce is sampled and from the garden or commercial plot at which farm stand produce collected as background/reference samples was grown. Each composite will consist of five discrete subsamples collected from the four corners and the center of the garden plot. The laboratory will split the samples. One split will be analyzed for dioxins/furans, PAHs, PCPs, arsenic, and TOC; the other will be archived for

future additional analyses, if necessary. Visual observations of garden soil characteristics—including soil texture, color, grain size, and presence of fertilizers and/or other soil amendments—will be noted in the field logbook. Characteristics of surface soils adjacent to each garden plot will also be noted.

Samples of produce similar to those collected from Area A residential gardens will be purchased from a local farm stand (if it can be documented that this produce was grown locally in an area unimpacted by the site) and submitted to the laboratory for analysis. The laboratory will split the samples. One split will be analyzed for dioxins/furans, PAHs, PCPs, and total arsenic; the other will be archived for future additional analyses, if necessary. Results of the off-site and unimpacted produce analyses will provide a reference point for COPC concentrations in produce grown outside of Area A.

2.1.5 House Dust

2.1.5.1 Objectives

House dust sampling will be performed to support the human health risk assessment. Residents are assumed to contact surface soil from their yards through incidental ingestion of soil tracked into homes by wind, people, and pets. This soil is present as house dust. Yards are frozen and/or covered with snow for a portion of the year, minimizing exposure to soils during this period; therefore, the incidental ingestion of house dust may be a more significant exposure pathway, relative to the outdoor soil contact exposure pathways, for residents of Area A.

2.1.5.2 Technical Approach

Indoor house dust samples will be collected from "high traffic" areas of residences in Area A. In 2001 and 2003, surface soil samples collected from residential yards in the vicinity of the St. Regis site were analyzed for metals, semivolatile organic compounds (SVOCs), volatile organic compounds (VOCs), dioxins/furans, and PAHs (Tetra Tech 2002). Ten residences, from this set of 20, will be sought to participate in the house dust study, yielding a total of 10 house dust samples. Residences will be identified prior to beginning fieldwork to reflect the range of dioxin/furan concentrations measured in soil samples collected in 2001 and 2003. Identified residences will include residences with low, moderate, and higher dioxin/furan concentrations based on soil samples collected in 2001. If a resident does not wish to participate in the sampling, an alternative residence with similar dioxin/furan concentrations (to the extent possible) will be selected.

Concentrations of dioxins/furans (as 2,3,7,8-TCDD TEQ), PAHs, and arsenic exceeded residential soil screening levels in some of the residential soil samples; therefore these chemicals will be analytes for house dust samples. Samples will be collected using a

vacuum sampler at all residences. Typical sample locations include the flooring directly inside the front door and in the main living areas. The sample locations will be a composite of two areas, each covering one square meter.

2.2 AQUATIC SAMPLING

The DQO process identified the need for additional sampling of sediments, benthic invertebrates, plant tissue (including wild rice), and surface water. The following sections present the objectives and technical approach for sampling of each aquatic matrix. Analyte lists and sampling areas for the aquatic sampling are summarized, by matrix, in Table 1. Proposed sample counts for the aquatic sampling are summarized in Table 2. Sampling methods and quality control are discussed in Section 3 and presented in detail in Appendices A and B.

2.2.1 Sediment

2.2.1.1 Objectives

The overall objective of the sediment investigation at the Site is to obtain sufficient data for ecological and human health risk assessments and to support remedial decision making. Data gaps for sediment were identified in Fox Creek, the Channel Area, Pike Bay, and in the background/ reference areas. These data gaps are described below for each of these areas.

2.2.1.1.1 Fox Creek

Existing data provide an incomplete understanding of the distribution and magnitude of COPEC concentrations in Fox Creek, as well as physiochemical characteristics such as TOC and grain size that affect COPEC bioavailability and toxicity. Also, results from split sediment samples collected in 2001 and analyzed by EPA and International Paper show order of magnitude differences in dioxin/furan TEQ concentrations. These differences appear to be related to differences in detected concentrations rather than differences in detection limits. These differences warrant further investigation of dioxin/furan concentrations in Fox Creek. Finally, total PCBs analyses were limited in the previous investigations of Fox Creek[4].

[4] All five sediment samples collected from the Fox Creek delta in 2001 were analyzed for total PCBs. However, only one sample was analyzed for PCBs in each of the two other sub-areas of Fox Creek, adjacent to the City Dump and adjacent to the Southwest Area. While the concentration of total PCBs in Fox Creek sediment sample SD-FCSW-01(adjacent to the Southwest Area) did not exceed the screening benchmark, the concentration of total PCBs in the sample adjacent to the City Dump (SD-FCCD-03) did exceed the screening benchmark.

2.2.1.1.2 Channel Area

As with Fox Creek, there is an incomplete understanding of the distribution and magnitude of COPECs, as well as general sediment characteristics such as grain size and organic carbon content[5] for the Channel Area. Further, not all sediment samples collected by EPA in 2001 were analyzed for dioxins/furans. (International Paper did not analyze any split samples from this area for dioxins.) Given that split results for dioxins/furans from other sampled areas showed order of magnitude differences in calculated dioxin/furan TEQ concentrations, it is recommended that additional sampling for dioxin/furan analysis be performed in the Channel Area.

No sediment samples have been collected at the south end of the Channel where it opens into Pike Bay. (Note: The surface water typically flows from Pike Bay to Cass Lake, but may periodically reverse due to dam operations and possible high river flows.) To complete the characterization of the Channel sediment, and to bound the understanding of the nature and extent of COPCs/COPECs, sampling in this area is recommended.

2.2.1.1.3 Pike Bay Shoreline

Sediment COPC/COPEC concentrations have not yet been characterized in the nearshore area of Pike Bay between the Fox Creek Delta. Therefore, sampling is proposed to determine if COPCs/COPECs are present in the area.

2.2.1.1.4 Background/Reference Areas

Chemical concentrations in Fox Creek sediments upstream of the Site may exceed concentrations found in other local background sources[6]. Collection of additional sediment samples upstream in Fox Creek is recommended to better characterize background/reference conditions.

[5] TOC was measured at each of the six historical sediment samples collected within the Channel and TOC content ranged from 12.8% (SD-HWY-01) to 30.1% (SD-HWY-02). Limited information on general sediment characteristics and grain size is available for the Channel. Based on the TOC data alone, the sediment environment in the Channel area may be more homogeneous than the sediments found in Fox Creek.

[6] Of the available site-specific background data collected as part of the St. Regis site investigation in 2001 (Tetra Tech 2002) a statistical comparison of the three creek and three lake sediment samples indicated that the concentration ranges were not significantly different (t-test; alpha = 0.05). Therefore, it was deemed appropriate to combine these data. The single reference upstream sediment sample in Fox Creek was not statistically compared to the other background data

2.2.1.2 Technical Approach

To fill these data gaps, the additional sediment sampling will be performed as specified below.

2.2.1.2.1 Fox Creek

A qualitative habitat/biota survey of Fox Creek will be performed prior to the onset of field sampling. The objectives of the survey will be to classify major habitats (open-water, wetland, marsh, etc.) and characterize the bank vegetation, emergent/submergent vegetation, substrate characteristics, surface water characteristics, and benthic animal community. This information will be used to identify exact sampling stations. The general sampling locations are shown in Figure 4. It should be noted that the survey of Fox Creek will be relatively basic and will focus on field conditions (including basic characterization of habitat and biota) at potential sample locations. Field conditions will be documented in photographs and logbook entries. EPA's rapid bioassessment protocols (Barbour et al. 1999) will be used as a guide for conducting the habitat survey. The survey will also include a qualitative benthic animal evaluation to determine if bivalves, crayfish, and aquatic insects are present for sampling, and qualitative field survey of benthic macroinvertebrates (presence and relative density of major taxa). Based upon results of the field survey and subsequent sediment/biota sampling, a more detailed survey may be considered at a later date. The results of the survey will be provided as soon as it is completed to the EPA oversight representative at the site. Final sampling locations will be approved by EPA.

The survey will evaluate general sampling locations to identify exact locations for collecting sediment and benthic invertebrate tissue data. Twenty-five sediment stations (0 – 20 cm depth) in Fox Creek will be targeted for sampling, six of which will be located at previously sampled sediment stations (see Figure 4). Station locations will span the entire length of the creek and will also consider transport and deposition of COPECs released from the former city sewage treatment plant and the City Dump Area. Ten sediment samples will be collected from wetland areas, 10 samples will be collected from open-water depositional areas, and 5 samples will be collected from open-water erosional areas. Benthic invertebrate tissue sampling locations will be among these 25 stations. The location of each sediment station may include a range of approximately 25 meters upstream and downstream of a fixed point depending on sediment and benthic invertebrate availability. Wet sieving methods will be used to obtain an estimate of grain size distribution of all sediment samples while in the field. Detailed sediment sampling procedures are specified in the FSP (Attachment A).

Fox Creek sediment samples will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides, TAL metals, grain size, and TOC. (Note: "dioxin-like"

PCB congeners will be determined in up to six of the archived samples. The samples selected for this additional analysis will be the samples that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). A split of all sediment samples that exceed screening levels for one or more COPECs[7] will also be submitted for sediment bioassay testing[8]. In addition, ammonia[9], a possible interfering toxicant, and acid volatile sulfide, which controls the toxicities of several heavy metals, will also be determined in the sediment samples submitted for toxicity testing. A portion of these samples will also be submitted for bioaccumulation testing and analysis using *Lumbriculus* (see Section 2.2.2.2).

2.2.1.2.2 Channel Area

A qualitative habitat/biota survey of the Channel area will be performed prior to the onset of field sampling as described above for Fox Creek. The survey objectives and procedures are described above in Section 2.2.1.2.1. Based upon results of the field survey and subsequent sediment/biota sampling, a more detailed survey may be considered at a later date.

Five sediment stations (0 to 20 cm depth) will be targeted for sampling in the Channel Area, three of which are previously sampled locations (see Figure 5). Samples will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides, TAL metals, grain size, and TOC. (Note: "dioxin-like" PCB congeners will be determined in one of the archived samples. The sample selected for this additional analysis will be the sample that exhibits the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). A split of

[7] The decision on whether to submit a sediment sample for bioassay analysis will depend on the results of the chemical testing for both COPCs and other analytes that could potentially confound bioassay result interpretation (i.e., total ammonia). Low screening benchmarks, such as those used in the RAWP, will be used to screen sediment sample results; high screening benchmarks may also be used. Sediment samples submitted for bioassay testing will be those that exceed either low or high screening chemical benchmarks. Selection of specific stations for bioassay testing will be decided prior to the lapse of the holding time on the archived samples. Archiving of sediment samples for potential bioassay testing will allow a holding time of up to eight (8) weeks.

[8] Sediment bioassay testing refers here to benthic invertebrate toxicity testing. This is a measure of the direct toxicity of sediments to benthic invertebrates.

[9] There are no historical data available for ammonia concentrations in sediment, but given the elevated levels of organic carbon in the sediments it is possible that ammonia is present at concentrations that are toxic to benthic invertebrates. Ammonia is produced from the natural decay of organic matter. In the event that ammonia is identified as a possible stressor, it may be necessary to remove ammonia from samples exhibiting toxicity and re-test them. If sample manipulation is believed to be necessary, EPA will be consulted prior to test initiation to determine appropriate sample manipulation procedures.

all sediment samples that exceed screening levels for one or more COPECs[7] will also be submitted for sediment bioassay testing[8]. In addition, ammonia, a possible interfering toxicant, and acid volatile sulfide, which controls the toxicities of several heavy metals, will also be determined in the sediment samples submitted for toxicity testing. A split of these samples will also be submitted for bioaccumulation testing and analysis using *Lumbriculus* (see Section 2.2.2.2).

Wet sieving methods will be used to obtain an estimate of grain size distribution of all sediment samples while in the field. Based on historical sediment organic carbon data, it is assumed that the sediment environment in the Channel Area is not as heterogeneous as the sediment environment in Fox Creek. Therefore, the proposed sediment sample collection does not distinguish between depositional samples and sandy/erosional samples.

2.2.1.2.3 Pike Bay Shoreline

Seven sediment stations (0 to 20 cm depth) in the shoreline area of Pike Bay are targeted for sampling (see Figure 5). Proposed sediment sample locations will be determined in the field and will be approved by and discussed with EPA. The sampling locations will be designed to provide coverage from the channel south approaching Fox Creek and include two locations adjacent to the existing swimming beach area at the city park and locations identified by the Band where reeds and other plants are gathered. In general, sediment samples will be collected from locations with water depths of between 2 to 4 feet to reflect those areas that may be contacted during human wading and swimming activities. These samples will be collected and analyzed with the primary intent of assessing the potential for human exposure to COPCs. The data will also be evaluated in the ecological risk assessment. Samples will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides, TAL metals, grain size, and TOC. (Note: "dioxin-like" PCB congeners will be determined in up to two of the archived samples. The samples selected for this additional analysis will be the samples that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). A split of all sediment samples that exceed screening levels for one or more COPECs[10] will also be submitted for sediment bioassay

[10] The decision on whether to submit a sediment sample for bioassay analysis will depend on the results of the chemical testing for both COPCs and other analytes that could potentially confound bioassay result interpretation (i.e., total ammonia). Low screening benchmarks, such as those used in the RAWP, will be used to screen sediment sample results; high screening benchmarks may also be used. Sediment samples submitted for bioassay testing will be those that exceed either low or high screening chemical benchmarks. Selection of specific stations for bioassay testing will be

testing[11]. In addition, ammonia[12], a possible interfering toxicant, and acid volatile sulfide, which controls the toxicities of several heavy metals, will also be determined in the sediment samples submitted for toxicity testing. A portion of these samples will also be submitted for bioaccumulation testing and analysis using *Lumbriculus* (see Section 2.2.2.2).

Wet sieving methods will be used to obtain an estimate of grain size distribution of all sediment samples while in the field.

2.2.1.2.4 Background/Reference Areas

Three background sediment samples are targeted for collection in upstream areas of Fox Creek and three background sediment samples are targeted in upstream wetland areas. The exact locations will need to insure that no potential contamination could have resulted from the transport of sediments upstream from the Site to the reference locations during storm events. The surface water typically flows from Pike Bay to Cass Lake, but may periodically reverse direction due to dam operations and high river flows. Samples will be analyzed for dioxins/furans, PAHs, PCBs (as Aroclors), chlorinated pesticides, TAL metals, grain size, TOC. (Note: "dioxin-like" PCB congeners will be determined in one of the archived samples. The sample selected for this additional analysis will be the sample that exhibits the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). A split of background sediment samples, which correspond to non-reference sediment samples submitted for bioassay testing, will also be submitted for bioassay testing[13]. A split of these samples will also be submitted for bioaccumulation testing and analysis using

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decided prior to the lapse of the holding time on the archived samples. Archiving of sediment samples for potential bioassay testing will allow a holding time of up to eight (8) weeks.

[11] Sediment bioassay testing refers here to benthic invertebrate toxicity testing. This is a measure of the direct toxicity of sediments to benthic invertebrates.

[12] There are no historical data available for ammonia concentrations in sediment, but given the elevated levels of organic carbon in the sediments it is possible that ammonia is present at concentrations that are toxic to benthic invertebrates. Ammonia is produced from the natural decay of organic matter. In the event that ammonia is identified as a possible stressor, it may be necessary to remove ammonia from samples exhibiting toxicity and re-test them. If sample manipulation is believed to be necessary, EPA will be consulted prior to test initiation to determine appropriate sample manipulation procedures.

[13] The 'corresponding' reference station will be selected to have similar grain size and TOC characteristics, but an absence of COPCs above screening levels. In the event that upstream site specific background locations in Fox Creek have COPC concentrations that make them unacceptable as reference stations, alternate reference station sediment will be analyzed. In this case, additional reference sediment samples will be collected from the reference creek sampled by EPA in 2001 (Tetra Tech 2002).

Lumbriculus (see Section 2.2.2.2). In addition, ammonia and acid volatile sulfide will also be determined in the sediment samples submitted for toxicity testing. Wet sieving methods will be used to obtain an estimate of grain size distribution of all sediment samples while in the field.

2.2.2 Benthic Invertebrates

2.2.2.1 Objectives

Invertebrates living in sediment in some areas of Area B are potentially exposed to elevated concentrations of COPECs and may accumulate these chemicals in their tissue. No data are available regarding COPEC concentrations in benthic invertebrates. This information is necessary to complete the exposure assessment for benthic invertebrates and wildlife that forage on them.

In addition, no data are available regarding potential direct effects to the benthic community resulting from exposure to COPECs. However, this data gap will be addressed by performing bioassay testing using select sediment samples (Section 2.2.1.2).

2.2.2.2 Technical Approach

Benthic invertebrate sampling is planned for Fox Creek, the Pike Bay Shoreline area, the Channel Area, and the background/reference areas. Bivalves and crayfish are the targeted species[14]. (Ingestion of aquatic insect larvae and nymphs may also be an important exposure route for mammals and birds. However, it is assumed that the available biomass is insufficient for the planned chemical analyses. Therefore, a laboratory bioaccumulation test with *Lumbriculus* will be performed as a surrogate.) The qualitative habitat/biota survey, described previously in Section 2.2.1.2, will also include a qualitative benthic animal evaluation to determine locations for collecting benthic invertebrates, and a qualitative field survey of benthic macroinvertebrates (presence and relative density of major taxa). Based upon results of the field survey and subsequent sediment/biota sampling, a more detailed survey may be considered at a later date.

The survey will identify exact sampling locations based on historical sampling locations, the presence and evidence of invertebrates, and other factors. The survey will target

[14] A survey by the Minnesota DNR (Helgen 1990) indicated that three crayfish species of the genus *Orconectes* were found in Cass County. The web site for the USDA Forest Service Chippewa National Forest indicates that four species of mussels are either common or abundant in the region (http://www.fs.fed.us/r9/chippewa/plan/aquatics/mussels.htm). Larval aquatic insects can make up a substantial portion of benthic invertebrate abundance. See also the invertebrate list presented in Appendix 7 of the Leech Lake Band of Ojibwe Pilot Superfund Project (LLBO 2003).

depositional and sandy/erosional areas in Fox Creek. Substrates in the other areas are less heterogeneous. The survey will identify locations for:

- Collecting crayfish and collocated sediment samples
- Performing caged bivalve bioaccumulation tests and collecting collocated sediment samples, and
- Collecting sediment samples that will be split for *Lumbriculus* bioaccumulation tests (to be performed according to EPA (2000) guidance)

If caged bivalve tests are not feasible, alternative methods for collecting these data, such as laboratory bioaccumulation tests, will be performed with the approval of U.S.EPA. The survey will determine whether one or both types of invertebrates are present in the sample area in order to establish the exact collocated invertebrate and sediment sample locations (see Section 2.2.1.2). Specific methods (e.g., test species, duration, etc.) for the bioaccumulation tests will be finalized with EPA, based on ASTM (2000) guidance.

Ten sampling stations will be selected from Fox Creek and 5 sampling stations will be selected from the Channel area. Three sampling stations each will be selected from Fox Creek and Fox Creek wetland reference areas. Fox Creek wetland will, however, collect only crayfish. From Pike Bay, 3 sampling stations for crayfish and bivalves and 5 stations for oligochaetes will be selected. All locations will be selected in consultation with EPA.

Crayfish, bivalve, and *Lumbriculus* tissue samples will be analyzed separately for dioxins/furans, PAHs, PCP, PCBs (as Aroclors), chlorinated pesticides, TAL metals, and lipids. (Note: "dioxin-like" PCB congeners will be determined in up to two of the archived samples. The samples selected for this additional analysis will be the samples that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). Finally, it should be noted that in addition to considering the invertebrate tissue sample analytical results, the ecological risk assessment will also consider estimated invertebrate tissue concentrations calculated based on site-specific sediment analytical results and bioaccumulation factors available in the literature and in EPA guidance.

2.2.2.3 Technical Approach for Benthic Invertebrate Toxicity Testing

As discussed in Section 2.2.1.2, sediment samples exceeding screening levels for one or more COPECs will be submitted for toxicity testing using the EPA amphipod survival and growth test and the EPA midge survival and growth test (EPA 2000). Control and reference sediment samples to evaluate will be discussed with EPA.

2.2.3 Plant Tissue

2.2.3.1 Objectives

Sampling of wild rice and other gathered plant tissues from Area B (and the forested wetland and Fox Creek) and a reference location will be performed to support the human health risk assessment. Ingestion of COPCs in wild rice collected from Pike Bay and the Channel Area and other gathered plants in these same areas, as well as the Forested Wetland and Fox Creek, are potentially complete, but uncertain, exposure pathways. Limited information is available regarding COPC concentrations in native plants. Sampling proposed here is intended to assess whether concentrations of potentially bioaccumulative COPCs in wild rice (growing in Pike Bay and the Channel area) and other gathered plants from these same areas, as well as the Forested Wetland and Fox Creek, are higher than off-site reference conditions and human health screening levels. The wild rice tissue data will also be used in the ecological risk assessment to evaluate risks to semi-aquatic herbivores such as muskrats and ducks.

2.2.3.2 Technical Approach

Plant tissue samples from a total of eight (8) locations will be collected in portions of Area B outside of the Southwest Area and the City Dump.

At these locations, plant tissue samples (gathered plants only) will be collected with composite soil and sediment samples. Soil samples will be collected in the Chippewa National Forest and the Forested Wetland, and sediment samples will be collected in the Channel Area, Pike Bay, and Fox Creek. The precise locations from which samples will be collected and the distribution between sediment and soil samples will be determined in the field in consultation with Leech Lake Band representatives and local biologists.

Wild rice samples will be collected from up to three Leech Lake Band harvest areas. The location(s) of the harvest area(s) will be determined prior to sampling by consultation with the EPA and the Leech Lake Band. Sample collection will be coordinated with gathering by Leech Lake Band members and will coincide with the peak harvest season in September. If less than three Leech Lake Band harvest areas are identified, multiple samples will be collected from the largest of the identified harvest beds such that a total of three wild rice samples are collected.

Because the rice requires finishing (i.e., parching, removing hulls, and winnowing) prior to human consumption, the most appropriate form of rice to evaluate in the human health risk assessment is processed rice. Therefore, one grab sample will be obtained from the rice harvest of each area after finishing by Leech Lake Band members, for a total of three samples of processed rice. Rice will be washed to reflect normal, everyday washing.

To evaluate any impacts the finishing process may have on the wild rice samples (and to better represent ecological receptor exposures), one grab sample also will be collected from each of the harvest areas prior to processing (unprocessed samples will include the seed, the protective sheath covering the seed, the stem, leaves, etc.), for a total of three samples of unprocessed rice. The grab sample submitted for analysis will also include young (immature) plants that are also eaten by wildlife.

One grab sample each of processed (if available) and unprocessed wild rice will also be collected from a reference harvest area(s). The reference area will be determined in coordination with the EPA and Leech Lake Band prior to sampling.

All wild rice samples will be analyzed for dioxins/furans, PAHs, PCBs (as Aroclors), PCP, and TAL metals. (Note: "dioxin-like" PCB congeners will be determined in up to one of the archived samples. The sample selected for this additional analysis will be the sample that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed). Information on sampling methods is presented in Attachment A.

Composite sediment samples from the 0- to 20-cm depth interval will be collected from each wild rice bed from which wild rice is sampled. Each composite will consist of five discrete subsamples collected from the perimeter (four subsamples) and the center (one subsample) of the rice bed. Sediment samples will be analyzed for dioxins/furans, PAHs, PCP, PCBs (as aroclors), TAL metals, grain size, and TOC. (Note: "dioxin-like" PCB congeners will be determined in up to one of the archived samples. The sample selected for this additional analysis will be the sample that exhibit the highest Aroclor concentrations. However, if Aroclor concentrations are not elevated, the congener analysis will not be performed).

2.2.4 Surface Water

2.2.4.1 Objectives

As mentioned in Section 2.2.1, concerns have been raised that hazardous substances originating from the St. Regis Paper Company Site may have resulted in impacts to the forested wetland immediately east of the Former Operating Area. Surface water in this area has not been sampled previously. Therefore, sampling is proposed to determine if COPCs/COPECs are present in the area above the human health and ecological screening levels identified in the RAWP.

2.2.4.2 Technical Approach

Collocated surface soil (see Section 2.1.1.2.3) and surface water samples will be collected from four (4) grid cells locations in the forested wetland. Surface water samples will be collected from the center of each grid cell (if no surface water is present at the center of a cell, surface water will be collected from one of the cell corners and documented). Samples will be analyzed for PAHs, PCP, TAL metals, and TSS. It should be noted that surface water samples will be collected from the mid-point of the water column at each location. The precise location and orientation of the four-cell grid shown will be finalized in the field based on the location of areas with collocated soil and surface water samples. Any changes will be discussed with and approved by EPA.

Analytical results will be screened in the risk assessment following the procedures described in the RAWP. If chemicals of potential concern are identified, the need for additional sampling and/or quantitative evaluation in the risk assessments will be assessed.

2.2.5 Fish Tissue

Numerous samples of game fish (perch, walleye, and lake whitefish) and white suckers were collected and analyzed from Pike Bay, Cass Lake, and the reference area in 2001 and 2002. International Paper's review of the laboratory data packages for these samples identified many issues related to analytical data quality. International Paper's technical concerns with the 2001 and 2002 fish tissue datasets were most recently documented in an April 5, 2004, letter from Tom Ross of International Paper to Timothy Drexler of EPA. To date, these issues remain unresolved.

EPA performed additional fish tissue sampling in Spring 2004. White sucker, walleye, and perch tissue and egg samples were collected from Pike Bay, Cass Lake, and Fox Creek Ecosystem and a reference lake (Bow String Lake). (Note: walleye eggs were not available from the reference lake). The table below shows the number of samples of each type that were collected. These samples were collected pursuant to the SAP for Fish Sampling Activities (Tetra Tech 2004b).

Fish Species	Size	Sample Type	Number of Samples Collected*	
			Cass Lake/Pike Bay Ecosystem	Bow String Lake
White sucker	NA	Eggs	5	3
Walleye	Large	Whole body	10	3
Walleye	NA	Eggs	4	0
Perch	NA	Eggs	2	3

Notes:
* = International Paper collected splits of all fish tissue samples.
NA = Not applicable

Additional fish tissue samples are required to fill identified data gaps (Tetra Tech 2004a) and to facilitate resolution of issues related to the quality and use of analytical results associated with previously collected fish tissue samples. Additional fish tissue samples will be collected by USEPA and the Leech Lake Band of Ojibwe in the Fall 2004 and analyzed by International Paper. As summarized below, samples will be collected from the Cass Lake, Pike Bay, and Fox Creek ecosystem and Bow String Lake and will include: lake whitefish eggs, whole body white suckers (both small and large fish), and lake whitefish fillets, whole body, and partial body (that portion of the fish remaining after the fillets have been removed) samples. As shown in the table below, some of the fish tissue samples will be archived. Archived fish tissue samples will be analyzed if analytical results for at least one COPC or COPEC exceeds a value equivalent to approximately two standard deviations greater than the mean concentration for that COPC or COPEC based on analytical results for the collected fish tissue samples minus the disputed sample #CL-WH-14. The decision to analyze archived fish tissue samples will be conducted on a species-specific basis.

Species	Size	Sample Type	Cass Lake, Pike Bay, and Fox Creek Ecosystem (Analyze/Archive)	Bow String Lake (Analyze/Archive)
Lake whitefish	Large	Fillet	15/5	5/0
	Large	Partial	5/5	5/0
	Small	Whole	5/5	0/0
	NA	Eggs	5/0	3/0
White sucker	Large	Whole	5/5	5/0
	Small	Whole	5/5	0/0
		TOTALS	40/25	18/0

Notes:
NA = Not applicable

Samples will be collected following methods described in Attachment A, and will be analyzed for dioxins/furans, PAHs, PCBs (as Aroclors and "dioxin-like" congeners), chlorinated pesticides, TAL metals, and lipid content.

3. METHODS AND QUALITY ASSURANCE

Field sampling activities, field screening, and laboratory analyses will be performed in accordance with the project FSP (Appendices A). Field quality control samples will include duplicate samples and field equipment rinsate blanks collected at an approximate frequency of one for every ten investigative samples. Approximately one of every twenty investigative samples will be designated as matrix spike/matrix spike duplicates. All field and laboratory data generated during the investigation will undergo data quality assurance review according to the procedures specified in the QAPP.

4. SCHEDULE

The table below outlines the duration of each project element and the completion deadlines.

Project Element	**Project Element Duration**	**Deadlines from Effective Date of Order**
Submit revised QAPP responding to USEPA comment letter of August 4, 2004 from Tim Drexler to Tom Ross.	10 days	10 days
Field mobilization for reconnaissance	2 weeks	2 weeks
Sampling	12 weeks	13 weeks after QAPP approval
Laboratory Analyses	6 weeks	19 weeks after QAPP approval
Data Quality Assurance Review	3 weeks	22 weeks after QAPP approval
Preparation and Submittal of Data Report (including validated data summary	6 weeks	28 weeks after QAPP approval

5. REFERENCES

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