MISSOURI
DEPARTMENT OF
NATURAL RESOURCES

Integrated Site Inspection/Removal Site Evaluation
Sampling and Analysis Plan

Shapiro Brothers Salvage Yard
Festus, MO
Jefferson County

Prepared By:

Missouri Department of Natural Resources
Division of Environmental Quality
Hazardous Waste and Environmental Services Programs

Table of Contents

1.0 Introduction 1
2.0 Site Information 1
2.1 Location 1
3.0 Data Quality Objectives 6
3.1 Problem Statement 7
3.2 Planning Team 7
3.3 Conceptual Site Model 7
3.4 Study Questions 9
3.5 Inputs to Study Questions 10
3.6 Study Boundary 11
3.7 Decision Rules 12
3.8 Tolerable Limits on Decision Error 14
3.9 Sampling Design 15
4.0 Field Activities 20
4.1 Sample Collection 21
4.1.1 Surface and Shallow Subsurface Soil Sampling 21
4.1.2 In-Situ XRF Analysis 22
4.1.3 Lead Paint Analysis 24
4.1.4 Surface Wipe Samples 24
4.1.5 Street Sweepings Sample 24
4.1.6 Surface Water and Sediment Sampling 25
4.2 Sampling Order 26
4.3 Sample Conditioning and Analysis 26
4.4 Number of Samples, and Container and Preservation 27
4.5 Chain-of-Custody 29
5.0 Quality Control 29
5.1 Field Decontamination 29
5.2 Quality Assurance/Quality Control (QA/QC) Samples 30
5.2.1 Equipment Rinsate Blank 30

5.2.2 Duplicate Surface Water Sample 30
5.2.3 Duplicate Sediment Sample 30
5.2.4 Replicate Field ICS 30
5.2.5 XRF Precision Samples 31
5.2.6 Laboratory Subsampling Replicates 32
5.2.7 Laboratory QC 32
6.0 Investigation Derived Wastes (IDW) Plan 32
7.0 Site Safety 32
8.0 Reporting 33

Appendix A – Health and Safety Plan
Appendix B – Field Sheets
Appendix C – XRF SOP

1.0 Introduction

As authorized under the federal Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986, the Missouri Department of Natural Resources (Department), Hazardous Waste Program (HWP), Site Assessment Unit (SAU) is conducting an Integrated Site Inspection/Removal Site Evaluation (SI/RSE) at the Shapiro Brothers Salvage Yard in Festus, MO.

The objective of this investigation is to determine whether the site warrants a removal action and to gather data necessary for the preparation of a preliminary site score under the Hazard Ranking System (HRS). The scope of the investigation will include collecting and analyzing surface soil, shallow subsurface soil, surface water, sediment, and potentially shallow groundwater, surface wipe, and street sweepings samples.

2.0 Site Information

2.1 Location

The site is located at the intersection of 12th Street and Vine in east-central Festus, MO. It is a rectangular 7-acre area, oriented primarily north-south, along the boundary that separates Festus and Crystal City, MO (red boundary in Figure 1).

Figure 1. Shapiro Brothers Facility, Festus, MO.

2.2 Description

Shapiro Brothers, Inc. has been used as metal scrap processing and recycling facility since the 1940s. They also offer demolition services, dismantle out of service rail cars, and offer transportation services to their clients for scrap metal materials. Materials brought to the facility are processed by cutting with a torch, or with a 450 horsepower shear or 3500 horsepower shredder. The metal is processed and compressed into sizes that are accepted by a steel mill for recycling. All non-metal items are separated out - this is called auto fluff- and taken to a sanitary landfill.

Most of the facility operations are conducted in unpaved gravel-covered areas, areas with exposed bare soil or soil mixed with gravel. During periods of wet soil conditions, trucks leaving the facility would likely have historically tracked mud through the streets on their way out of the facility. There are two gates into the facility; a north gate and a south gate. Most of the traffic into and out of the facility occurs through the south gate, although fluff pile material is loaded onto trucks near the northern end of the facility which may also exit through the northern gate. A truck wash system was installed in the fall of 2011 to clean the undercarriage and wheels of trucks leaving the southern gate. The facility recently started operating a street sweeper in the roads around the facility to remove residual material tracked into the streets by trucks.

Prior to the spring of 2011, trucks primarily approached the facility by traveling along 12th Street to and from Truman Blvd. (U.S. Highway 61). A heavy spring flood event in 2011 washed out the culvert beneath 12th Street near the facility making the road impassable. Since then, trucks have traveled south from the facility on the residential street Delmar Ave. before connecting with Truman Blvd. via 6th Street.

Runoff from the facility enters a drainage that flows south to Plattin Creek. The drainage carries stormwater runoff and is carried by underground conduit resurfacing in various areas along its

route to Plattin Creek. Figure 2 shows the route of the drainage between where it begins just north of the facility to where it discharged into Plattin Creek near the waste water treatment plant 2 miles downstream. Plattin Creek then flows for about 1.8 miles to its mouth at the Mississippi. River.

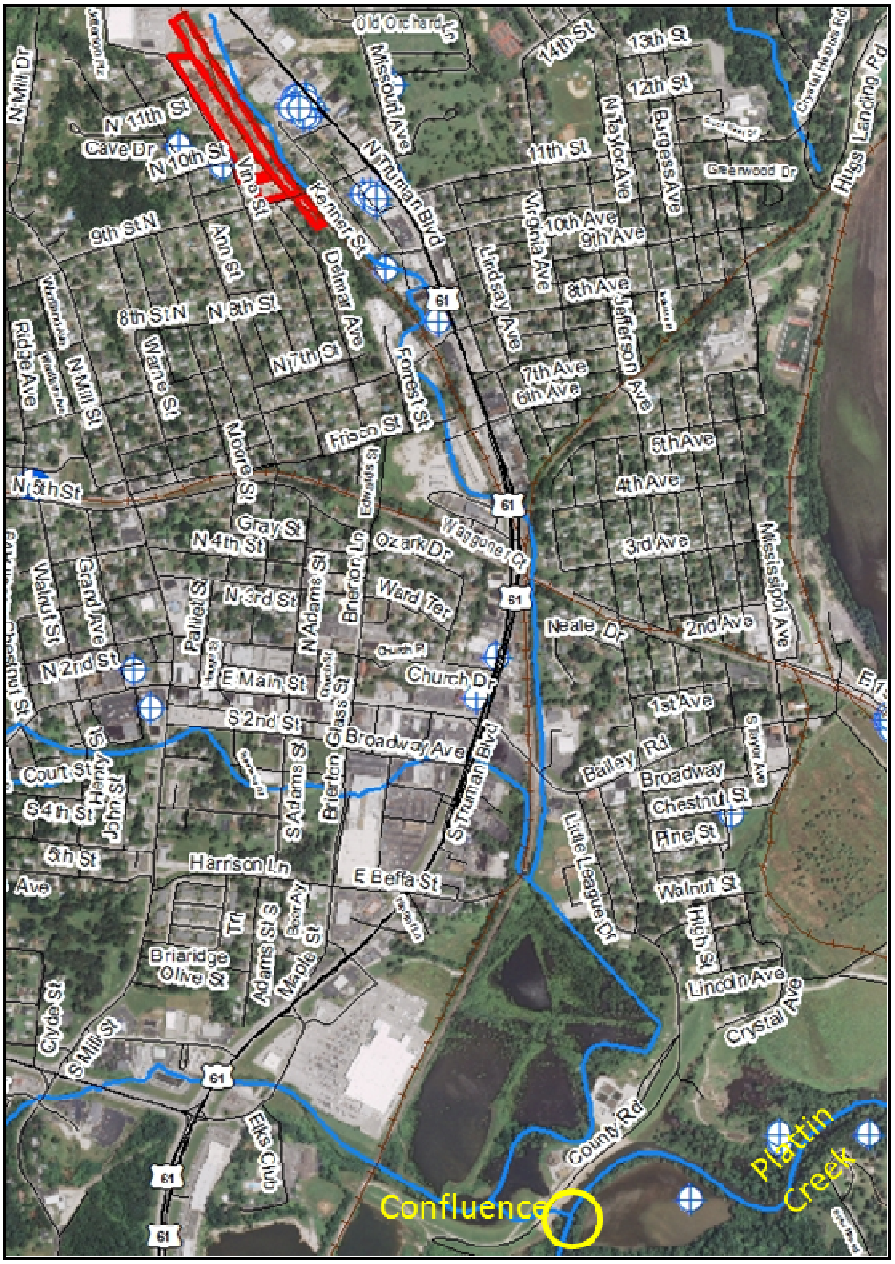

Figure 2. Surface Water/Storm Water Drainage

2.3 History/Contaminants of Concern

The site came to the departments' attention following complaints made to the City of Festus by nearby residents regarding the dust created at the facility and by trucks hauling material in and out of the facility. The department conducted a facility inspection in September 2011 and identified violations of hazardous waste management and water pollution laws (MDNR, 2011a, MDNR, 2011b, MDNR, 2011c). The facility is working with the department to address these violations. During the September 2011 inspection, surface soil samples were collected from various areas in the facility, and a sample of the truck wash solids was collected from the newly installed system at the south gate. Soil samples were analyzed for the eight RCRA metals. Elevated levels of lead, cadmium and other metals were found in every soil sample collected as well as in the truck wash solids. The city of Festus and a contractor working for Shapiro Brothers both collected separate samples of street sweeper debris from roadways surrounding the site (9th, Delmar, and Vine) in 2011 (Festus, 2011and NPN, 2011). Results for both samples show elevated levels of cadmium, lead and other metals in similar proportions to those found in facility soils and truck wash solids. The sample collected by the facility contractor contained over twice as much lead as the sample collected by the City. However, lead in the City's sample was found to be more leachable and would classify the street sweepings as hazardous waste under state and federal law.

Samples of surface water from upgradient and dowgradient of the facility were also collected during the September inspection. Results documented discharge of lead and cadmium in surface water runoff from the facility at levels in violation of state and federal law. No sediment sampling was conducted in the stream running north to south along the facility's eastern boundary. However, metal debris obviously originating from metal processing operations at the site was observed in the creek bed all along this stretch of the creek.

Department staff returned to the facility in January 2012 to resample the truck wash solids and street sweepings for waste characterization confirmation. Based on 3 samples, the truck wash solids waste, while containing lead levels up to 3,980 mg/kg, was characterized as non-hazardous under RCRA. A sample of the street sweepings containing 2,400 mg/kg lead was also characterized as non-hazardous waste.

Staff also collected soil samples at the facility to be analyzed for a broader range of potential contaminants. Results from those samples show that in addition to lead, the metals arsenic, cadmium, chromium, copper, nickel and zinc are present at elevated levels (MDNR, 2012). Other non-metal contaminants detected include PCBs, PAHs, phthalates, toxaphene, dioxins/furans and 4-chloro-3-methyl phenol. However, the concentrations of these other contaminants relative to commonly used soil screening levels are not nearly as significantly elevated as for lead. Although not analyzed for in the facility soil, polybrominated biphenyl ethers (PBDEs) may be of potential concern. PBDEs are flame retardant chemicals added to plastics, upholstery coatings and urethane foams such as would be expected to be present in the auto fluff material. There is growing evidence that PBDEs persist in the environment, accumulate in living organisms and may be associated with liver, thyroid and developmental toxicity (http://www.epa.gov/oppt/pbde/).

3.0 Data Quality Objectives

To help ensure precise, accurate, representative, complete, and comparable data, all field work and analyses will be conducted in accordance with the Quality Assurance Project Plan (QAPP) for Pre-Remedial/Pre-Removal and Targeted Brownfields Site Assessments Revision 6, December 7, 2007, and ongoing (MDNR, 2007). The QAPP describes the general data quality

objectives (DQO) for site assessment investigations conducted by the HWP and ESP. Those DQOs specific to this project are described below.

3.1 Problem Statement

Decades of metal shredding, cutting, processing, and hauling have resulted in releases of various heavy metals and other contaminants to surface soil at the facility. Some of these metals have also been documented in surface water discharging from the facility. The facility is located within a densely populated urban residential area, and nearby residents are concerned about migration of contaminants from the site to residential areas through tracking of contaminated soil on truck tires and via wind deposition.

3.2 Planning Team

The planning team includes staff from the HWP Superfund Section, EPA Region 7, ESP Field Services and Chemical Analysis Sections, USGS Columbia Environmental Research Center, Missouri Department of Health and Senior Services (DHSS) and Jefferson County Health Department risk assessors.

3.3 Conceptual Site Model

Elevated levels of arsenic, cadmium, copper, lead, nickel, and zinc have been measured in surface and shallow subsurface soil at the facility. Those same metals have also been measured in solids collected from the truck washing station near the facility entrance and in street sweepings samples collected from residential roads adjacent to the facility. Based on a comparison of the concentrations measured relative to common screening benchmarks, it is clear that the primary contaminant of concern for the site will be lead. Levels of lead exceeding 37,000 mg/kg have been measured in surface soil at the facility, and concentrations up to 4,800 mg/kg of lead has been measured in street sweepings collected from residential streets nearby. These levels far exceed most screening benchmarks for lead in residential yards which are in the range of 260-400 mg/kg.

Site-related contaminants can be released in a number of ways. Fine particulates from contaminated soils disturbed at the facility or from the material processing activities themselves could be transported via wind. Contaminated soils clinging to trucks leaving the facility could be released to roadways in residential areas where it could be transported as dust, or with runoff following rain events. Fine particulates released from the facility could be deposited on yard soil, on various exterior surfaces, or be transported into residences through open windows or via tracking in on shoes or other means. Residents could be exposed to contaminated particulates through direct contact with soil, dust on surfaces, or inhalation of particulates in ambient air.

Contaminants deposited on residential yard soils through air deposition would be expected to show a spatial pattern with higher concentrations nearer to the facility/haul road. Lead poses some unique challenges since it may also be present near residential structures due to past use of lead paint, and near roadways due to fallout from vehicle exhaust during the period when leaded fuel was used. The nation's only primary lead smelter is located approximately 2 miles north of the Shapiro facility and may be an additional source of lead contamination due to air deposition from the smelter or from the past use of smelter slag as traction control on streets of Festus in the winter.

Contaminants could be released from the facility to downstream surface water and sediments in a drainage leading to Plattin Creek and to Plattin Creek itself. Human exposure could occur with direct contact to stream water and sediment or through ingestion of fish living in contaminated stream reaches.

Metal containers (drums, tanks, railcars, gas tanks) which previously held solvents or other liquid chemical products may have been processed at the facility in the past. Should such containers have held residual product, it may have been released to the surface during the processing of the

containers. Historical releases of residual solvents may migrate vertically through soil contaminating shallow groundwater. Although this groundwater is not used as a drinking water resource in the immediate area, solvent vapors from contaminated groundwater may migrate and result in vapor intrusion into buildings near the site. Although this is a potential pathway of concern, it is considered a lower priority and sampling for this pathway will not be included in this phase of the investigation.

Decision Units

Each residential yard is a separate exposure unit (EU). CERCLA removal and remedial action decisions for residential yards are typically conducted on an EU by EU basis. Therefore, yard decision units (DUs) for this investigation will be set the same as the exposure units (the entire yard). However, to aid in evaluating source attribution and to evaluate sub-areas within the yard DUs that may have special exposure concerns (e.g. children's play areas), sampling will be conducted in sampling units (SUs) smaller than the DU. The sampling design is further discussed in Section 3.9.

3.4 Study Questions

The primary study questions for this investigation are:

- What is the background concentration of contaminants of concern (COCs) in residential yards unaffected by facility operations?
- Do mean concentrations of COCs in the fine fraction of surface soil (0"-2") in residential yards DUs adjacent to the facility or along haul routes significantly exceed background levels and/or health-based screening levels?
- Are COCs in residential yard soils near the facility and along truck haul routes attributable to the facility operations?
- Do fine particulates on exterior residential surfaces contain COCs at concentrations above health-based screening levels?

- How far downstream from the facility do impacts (concentrations above water quality standards or sediment quality guidelines) extend?

3.5 Inputs to Study Questions

The following lists the primary inputs required to address the principal study questions.

- Screening Levels
  Obtained from a variety of sources including the Superfund Chemical Data Matrix, EPA Regional Screening Levels, Missouri Risk Based Corrective Action Guidance, Missouri Water Quality Standards, and USGS consensus based Sediment Quality Guidelines. Generally removal decisions for lead in residential yards are based on the EPA non time-critical removal action level (RAL) of 400 mg/kg and time-critical RAL of 1,200 mg/kg. No screening level is available for lead in dust on exterior surfaces. Instead, wipe sample results will be compared to benchmarks for interior floor surfaces from Section 403 of the Toxic Substances Control Act (TSCA) of 40 ug/ft^2. No readily avialable screening levels were identified for PBDEs in soil for a residential use setting.

- Estimates of mean lead concentrations
  - In the fine fraction (<0.25 mm) of soil from the top two inches in background DUs established in three city parks
  - In the fine fraction (<0.25 mm) of soil from the top two inches of SUs and DUs established in residential yards
  - In street sweepings collected from non-haul route roads
  - In fine particulates accumulated on various surfaces at residential properties

- Estimates of variability in lead concentrations in each SU and DU sampled

- Ratios of lead to zinc, arsenic, and cadmium in soil samples collected from the facility and from residential yards and street sweepings.

- Estimates of site-related contaminant concentrations in surface water at various distances downstream of the facility

- Estimates of site-related contaminant concentrations in sediment (<2mm particle fraction) at various distances downstream of the facility.

3.6 Study Boundary

The site consists of the facility itself plus residential properties, surface water and sediments affected by site activities. Since it is not known how far beyond the facility, if at all, site-related contamination extends, depending on the findings of this phase of the investigation, it may be necessary expand the investigation to include additional residences or stream segments. Access was requested from approximately 110 residential property owners who own property adjacent to the facility or the main haul roads. This figure also includes requests to owners in an area further away from the facility expected to be outside the influence of site contaminants (background area). Figure 3 shows the locations of each residential property for which access was requested as parcels with yellow borders. Access was granted by 70 property owners. However, approximately 20 of these were for properties in the background area, and only a small number of them are needed for the investigation. Therefore the number of properties we anticipate to actually sample is likely closer to 50-55.

Figure 3. Properties Identified for Potential Sampling

3.7 Decision Rules

Investigation data will be used to make decisions for both the Site Inspection (HRS scoring) and to assess the need for a Removal Action. Decision rules regarding HRS scoring are well described in the HRS Rule and HRS Guidance Document (USEPA, 1990 and USEPA, 1992). Decision rules for the Removal Action are described below. For both HRS and Removal Action purposes it is important to demonstrate that contamination is attributable to the source area (facility). This determination will be made based on a weight of evidence approach. The following observations will support attribution of contaminants to the facility.

<u>Attribution</u>

- Observation of significantly higher lead concentrations in street sweepings collected from near the facility and along haul roads (Delmar St.) as compared with those collected from residential streets further away.

- Observation of trend with higher lead concentrations in SU1 compared with those in SU2 at target residences.

- Observation of significantly lower trend or absence of trend in lead concentration between SU1 and SU2 at background residences.

- Similar ratios of lead to zinc, arsenic and/or cadmium in samples from residential yards, street sweepings collected near the facility and soil samples collected at the facility.

- Significant difference in lead concentration on wipe samples collected at targeted residences vs. background residences.

- Significant difference in lead concentrations of street sweepings collected from roads near the facility vs. from further away.

Removal Assessment

- If the estimate of the mean lead concentration in yard DU exceeds the EPA RAL of 400 mg/kg or 1,200 mg/kg and is attributable to the facility, a time critical or non-time critical removal action will be recommended; otherwise no further action will be taken for that residence.

- If the estimate of mean lead concentration in a special use SU (e.g. play area) exceeds the EPA RALs or lead on a surface wipe sample exceeds applicable criteria, the residence will be referred to EPA or DHSS risk assessors for a health consult to determine future course of action.

- If PBDEs are detected in yard soils at levels significantly higher than background levels, the results will be referred to EPA or DHSS risk assessors for a health consult to determine a future course of action.

3.8 Tolerable Limits on Decision Error

Our hypothesis is that yard soil at residences near the facility and along haul routes have site-related contamination at concentrations above health-based screening levels. Falsely rejecting that hypothesis, considered a Type I decision error, would mean mistakenly concluding that yards are clean. Falsely accepting this hypothesis, considered the Type II decision error, would mean concluding that the yards are contaminated when in fact they are not. The Type I error would result in the taking no action at yards actually contaminated at levels that could pose a

health threat to residents. A Type II error would result in the unnecessary use of resources to conduct removal and/or removal action at yards that do not warrant it. The Type I error is considered more severe since it results in potential threats to human health.

A sampling design has been chosen to control error and minimize the likelihood of making a Type I decision error. In-situ XRF analyses will be used to obtain a high density data set about concentration gradients for lead with distance from each residence. The use of field analytical techniques such as XRF allows much higher density data sets than is practical with conventional discrete soil sample/laboratory analysis. Large data sets will increase confidence about conclusions drawn from the data and decrease the likelihood of committing either type of error.

An incremental composite sampling (ICS) approach will be used to collect soil samples in each SU. ICS is designed to obtain single soil sample that contain contaminants in the same proportion in the sample as they are present in the SU (e.g. are representative samples). This is achieved through inclusion of many increments of adequate-mass soil across each SU. Representative sampling will decrease the likelihood of committing either type of error.

The ICS sampling procedure will be replicated and results of the replicates will be used to provide a conservative estimate (95% UCL) of the true mean lead concentration. By using a conservative estimate of mean concentration, we will protect against underestimating the true mean, and therefore potentially walking away from a residence that is actually contaminated.

3.9 Sampling Design

Based on the CSM, higher contaminant concentrations are expected closest to the suspected source of contamination (the facility or the haul road). This assumption will be tested by conducting in-situ XRF analyses along transects perpendicular to the suspected source. Several

transects will be set up at each residence, and 3-5 30-second in-situ XRF readings will be collected at several distances along each transect. At some residences, the XRF results will be used to identify a sampling unit (SU1) as a rectangle parallel to the road across the entire yard. For residences whose back yards are adjacent to the facility (e.g. those along Kenner Street), SU1 will be established parallel to the facility across the back of the property. Dimensions of this SU will depend on yard size and configuration and observed concentration trends from in-situ XRF analysis.

The use of SU1 is primarily to provide a line of evidence for attribution of contaminants to the suspected sources. Therefore, it is not necessary for to establish and sample a SU1 in each yard; only a subset of yards. For yards where in-situ XRF is conducted, but no SU1 is established, a single 30-increment ICS will be collected from the entire yard (excluding the drip zone DU as discussed below). For those residences at which SU1 is established, the area of the SU in square feet (ft^2) will be recorded on the field sheet, and a 16-increment ICS will be collected from this SU. Fewer increments are justified for SU1 since no cleanup decision is being made on the result; it is only being used in conjunction with the in-situ XRF analyses as an aid in attribution. Therefore a less accurate and precise estimate of the mean concentration in the SU is tolerable.

When SU1 is used at a property, a second SU (SU 2) will be established as the rest of the yard (excluding SU1 and the drip zone SU3) as shown in Figure 4. A 25-increment ICS will be collected from this SU. The area of SU2 will be measured using GIS tools after the field work is complete. If a special use exposure area is observed in a yard such as a children's play area, its area in ft^2 will be recorded on the field sheet and it will be sampled as a separate SU. The number of increments collected from a special use are will be determined in the field based on the SU size, but will contain no less than 15 increments. When an SU1 is not established at a yard, the entire yard (excluding the drip zone SU) will be sampled as a single decision unit with a 30-increment ICS.

Most of the homes in the study area were built prior to the banning of lead-based paint in 1978. Therefore it is possible that lead levels in soil near the structures may be elevated due to that potential source rather than to activities attributable to Shapiro Brothers Inc. This will be assessed through in-situ XRF analysis conducted along transects extending away from the house in 4 directions. This approach was used recently for a lead investigation in an older residential area of Kansas City, and showed that drip zone lead effects can extend out 6 feet or greater. The drip zone transects will be conducted at both the background and target residences.

Each SU and DU ICS will be air dried, disaggregated, sieved, and analyzed by XRF. For yards where an SU1 and SU2 sample are collected, an estimate of the contaminant concentration for the entire DU (yard) will be made by mathematically combining results for the ICS collected in SUs 1 and 2. A weight-averaging approach will be used using the formula below, where *i* is the number of SU-ICS (usually 2), *w* is the relative size of the region represented by each SU-ICS sample, and $\overline{X}$ is the mean of each ICS representing each SU region.

$$\text{Weighted Mean} = \sum_i w_i \overline{x_i}$$

Alternatively, this weight-averaging may be done by physically combining representative portions of each ICS sample and conducting an additional analysis on the combined sample.

An observation of concentration trends along the transects perpendicular to the suspected contaminant sources and decreasing contaminant concentrations from SU1→ SU2 will support the CSM and attribution of yard contaminants to facility activities. An observation that this trend is significantly lower or absent in yards of homes further from the facility (background yards) compared with those adjacent to the facility or along haul routes will also support attribution of

contaminants to the facility operations. Comparison of street sweeper debris collected from streets near the site with debris from streets outside the influence of the site will provide an additional line of evidence towards attribution. Results that show higher site-related contaminant concentrations in street sweepings collected closer to the facility would support attribution of yard contaminants to the facility. Results showing higher contaminant concentrations in wipe samples collected from similar surfaces at background residences vs. residences adjacent to the facility or to haul roads will further aid in attribution.

Based on the CSM, it is assumed that the release mechanism is similar for the yards, and therefore we expect similar levels of contamination and contaminant variability within the SU types across different yards. Therefore, rather than collected replicate ICS in each SU and DU to measure variability, we will collect 3 replicate ICS in 20% of the SUs. The replicate data from each SU type and DU will then be examined using graphical and statistical tools. If deemed appropriate, variability will then be extrapolated from DUs with replicate samples to those without for purposes of estimating uncertainty and upper confidence limits.

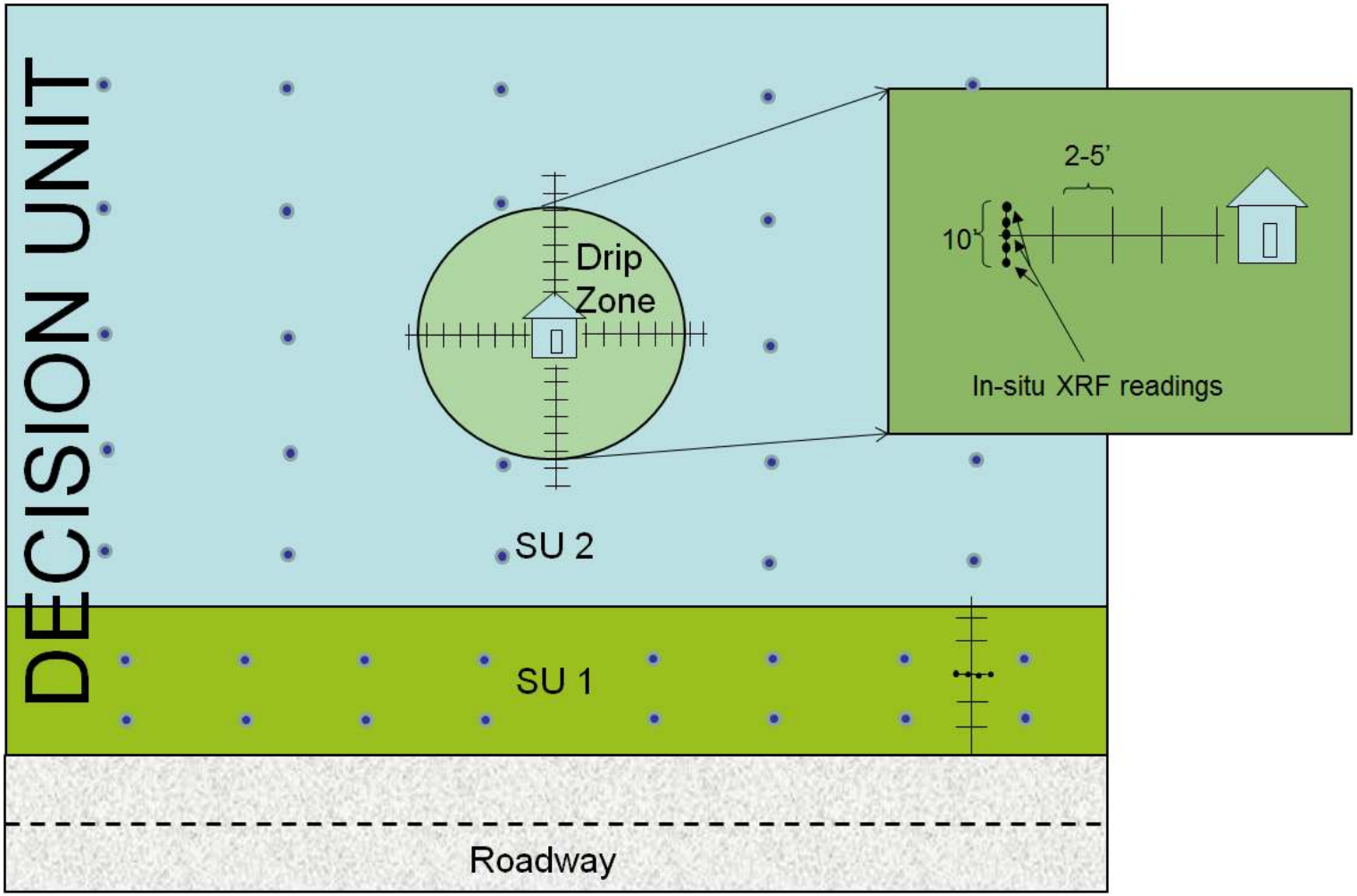

Figure 4. Sampling Design

4.0 Field Activities

The sampling event will include collection of surface and shallow subsurface samples at residential yards for which the department gains access. Approximately 70 residents provided access, but 20 of these were background residences. Since only 3-4 background residences are needed for the investigation, the total number of houses to be sampled is approximately 55. If staff are not able to complete sampling at all these residences in the 4 days planned, yards nearest the facility will be prioritized. Door hang tags will be prepared to place on doors of residences where access was provided but are not sampled.

Surface water and sediment samples will be collected upstream and downstream of the facility to near where the drainage enters Plattin Creek. No samples are planned from the stretch of Plattin Creek between the tributary mouth and the Mississippi River since the location where the drainage enters Platting Creek is at the same place where the city WWTP discharges and it would be difficult to decipher attribution between these two contaminant sources.

Surface wipe samples may be collected on various structural surfaces at some of the background and target residences based on field observations. A 30-increment composite sample will be collected from street sweeper debris collected by the City of Festus in areas outside the influence of the facility. All sample locations and descriptions and field notes will be recorded on field sheets forms shown in Appendix B. Personnel will determine global positioning system (GPS) coordinates of all surface water and sediment sampling locations. GPS locations will not be collected at yards since locational data for each parcel is already available from the county tax assessor. Site sketches and in-situ XRF readings at each residence will be recorded on the field sheets, and all samples collected will be recorded on chain of custody forms. Photographs will be taken to document various aspects of the sampling event.

4.1 Sample Collection

Except as otherwise noted, all aspects of sampling shall be performed using standard operating procedures (SOPs) established within the ESP, Environmental Emergency Response/Field Services Section (EER/FSS) for the collection, preservation, and transport of various media sampled. Modifications to the following sampling methods are described below and others may be made in the field based upon conditions encountered. Any modifications to the methods will be noted on the field sheets and in the SI/RSE Report prepared by SAU.

4.1.1 Surface and Shallow Subsurface Soil Sampling

In-situ XRF analyses will be conducted along transects set up perpendicular to the road to aid in attribution and to help establish the width of SU2. The boundary of SU2 will be set at a distance where distinct drops in lead concentration are observed with distance from the road. If trends in this distance are observed after several residences, in-situ XRF analysis will be discontinued and the width of SU2 will be set based on previous observations. The area of SU1 will be measured and recorded on the field sheet.

Surface soil ICS samples will be collected as follows. An EVS™ or EnviroStat™ stainless steel incremental sampling tool will be used to collect equal-mass increments of soil at equal spacing across each SU. A 2-cm diameter, 6-cm long stainless steel sampling core will be used. The sampling core will be advanced into the soil and ejected into a 2-gallon size heavy duty sealable plastic bag. This process will be repeated at each increment collection location, and all increments will be combined together into one bag. Bags will be labeled with the Location ID, SU#, date, time, & sampler's initials.

Past experience has indicated that drip zone lead effects can extend out 6 feet or more from residential structures. For this study in-situ XRF analyses will also be used initially to establish

the boundaries of the drip zone SU. Transects will be set up extending out from each side of the residence. Along each transect, 30-second in-situ XRF readings will be taken at 1'-5' intervals starting at 30" from the house and extending outward until a distinct significant drop in the lead concentration is observed or until the levels drop near background. At each distance interval, five separate in-situ XRF readings will be taken at 2' intervals perpendicular to the transect as shown in Figure 4. The average of the five readings will be used to represent the concentration for that distance interval. The estimated background level will be based on in-situ XRF readings collected from surface soil in West City, Sunset, and Billy Porter Memorial Parks. The distances that elevated lead levels extend away from a house will be used to construct a "drip zone" shown in Figure 4. If, after several houses a trend is observed in the extent of drip zone influences, in-situ XRF analyses may be ceased and the drip zone size inferred from previous observations.

ICS samples will be conditioned as described in Section 4.3 and analyzed for lead by XRF. A portion of the samples will also be submitted for laboratory confirmation analysis. A limited number of ICS samples collected from yards nearest the facility will be submitted for analysis of PBDEs, PAHs, Toxaphene and PCBs. An archived soil sample collected from beneath the auto fluff pile in January will also be submitted for analysis of PBDEs.

4.1.2 In-Situ XRF Analysis

The XRF analyzer will be calibrated and standardized as per the manufacturer's instruction. Known reference standards containing certified concentrations of lead at various levels will be analyzed prior to initiating field work. Results will be documented on an XRF data field sheet for each residence. The serial number of the analyzer(s) used at each residence will be noted on the field sheet. Prior to XRF analysis, the Location ID, SU, transect, distance & analyst will be entered into the XRF analyzer.

The average lot size of the residences selected for sampling is approximately 10,500 ft^2 (~ ¼ acre) An area of this approximate size will be established as background DUs in Sunset, West City, and Billy Porter Memorial Parks. Surface soil ICS samples will be collected from these areas as described in 4.1.1. The bags of soil from the background DUs will then be manually mixed to homogenize the soil as well as possible, and then XRF analysis will be conducted on the soil through the bags 5 times with mixing in between. The average of the 5 readings will provide an estimate of the background lead concentration in each background park DU. The lead result for each background DU will be averaged, and that value will be used as reference for in-situ measurements collected at target residences as described below. The collected bags of background soil will also be returned to the lab for processing and analysis as described in 4.4.

At each distance interval from the house along the transects, the surface vegetation will be removed. Any soil clinging to the vegetation roots will be shaken back out over the bare soil. The soil will be flattened out, large debris (rocks sticks) removed, and a 30 second in-situ analysis will be performed. The Location ID and SU# will be entered into the XRF prior to each analysis. The results will be recorded on the field sheet. Two to four additional in-situ analyses will be conducted at the same distance interval perpendicular to the transect at 2' spacings. The results will be recorded on the field sheet, and the averages for each distance interval will be calculated.

When a distinct drop is observed in the average lead concentration along a transect, no further in-situ readings will be taken for that transect, and a flag will be placed to mark the boundary of the SU for that transect. For the drip zone, the process will be repeated for the other transects to form a polygon around the house considered the "drip zone" of influence from lead-based paint.

In-situ XRF precision will be evaluated once per day per XRF analyzer by collecting 7 replicate XRF readings at a single location without moving the analyzer.

The XRF data will be downloaded from each analyzer upon returning from the field and will be QC-checked and validated.

4.1.3 Lead Paint Analysis

Accessible painted exterior surfaces at some residences built prior to 1978 may be analyzed for the presence of lead-based paint using an XRF analyzer. Results will be recorded on the field sheet. If a sufficient mass of paint flakes are obtainable on the soil surface near the residence, it may be collected in a baggie for potential analysis. Data from these analyses would be used as additional lines of evidence that lead detected in drip zones is due at least in part to lead paint.

4.1.4 Surface Wipe Samples

Wipe samples will be collected at some residences based on visual observation, proximity to the facility, and weather conditions. Wipe samples will be collected on 1 ft^2 surface areas identified in the field following the procedure outlined in the DHSS Lead Poison Prevention Manual (MDHSS, 2009). Surface wipes will be collected on comparable surfaces at both background residences and target residences. For example, if a surface wipe is collected from the east-facing exterior siding of a near-facility residential house, one will also be collected from the east-facing siding of a background residential house.

4.1.5 Street Sweepings Sample

The City of Festus will provide the Department with access to street sweeper debris collected in roadways located away from the Shapiro facility. A 30-increment composite sample will be collected from equally spaced locations across the collected street sweepings as is practical.

4.1.6 Surface Water and Sediment Sampling

Surface water grab samples will be collected by immersing the sample containers directly into the drainage leading to Plattin Creek in areas where it is accessible. Samples will be collected from locations upstream of the site, at probable points of entry of any runoff from the site, and from locations downstream of the site. Approximate sampling locations are shown in Figure 5. If required to enter water, personnel will approach the sampling location from downstream to minimize sediment disturbance during collection.

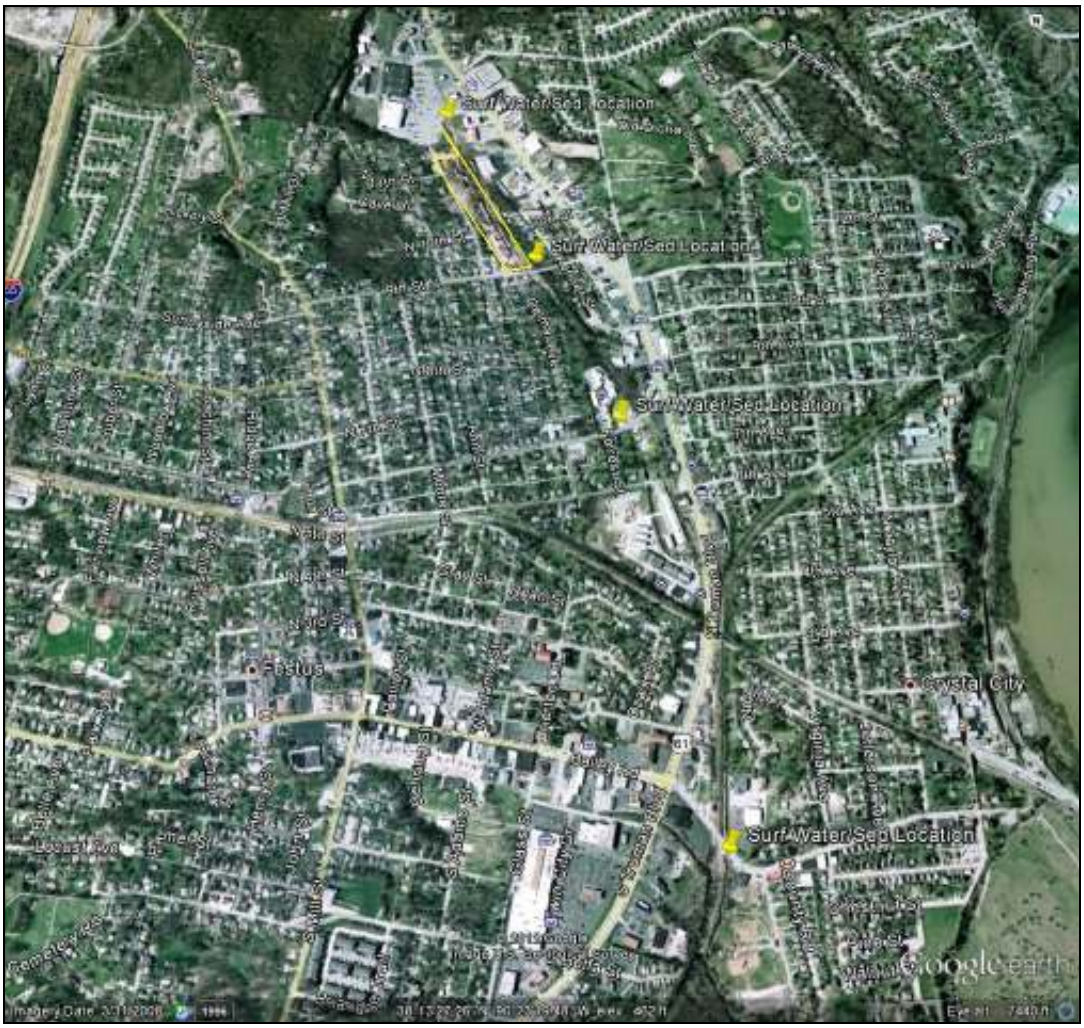

Figure 5. Approximate Surface Water/Sediment Sampling Locations

Sediment grab samples will be collected using clean stainless steel spoons to retrieve sediment from the 0- to 2-inch depth from the bottom of drainage. At each sediment sampling location, 15 increments of approximately 30 grams each will be collected along a transect perpendicular to the drainage. The increments will be combined to form a composite sample that represents average conditions of the sediment bed at that location. The increments of sediment will be transferred to clean aluminum foil pans and any excess water decanted. The sediment will be mixed thoroughly using a stainless steel spoon and placed into a resealable plastic bag. Sediment samples will be collected at the same general locations as surface waters.

4.2 Sampling Order

Though not always practical, attempts will be made to collect all samples in the order from least-to-most contaminated.

4.3 Sample Conditioning and Analysis

Soil and sediment samples will be returned to the laboratory and air dried in aluminum pans lined with wax paper. Since indoor dust is known to be a significant source of PBDEs, the samples to be submitted for that analysis will be dried and processed under a hood to minimize potential for contamination of the samples. The air dried samples will be returned to their original bags, placed inside an additional bag, and then disaggregated by striking the sample 30 times with a mallet to disaggregate clumps of soil. The soil sample will then be passed through a 0.25mm sieve to obtain the target particle size. The soil passing through the sieve will be placed inside a thin-walled resealable plastic baggie. XRF analysis will be conducted on the dried/sieved soil samples following the SOP in Appendix C. The XRF analysis of the SU ICS

samples for lead is primarily to provide a line of evidence for attribution of yard contaminants to roadway sources. After XRF analysis, representative area-weighted portions of the ICS collected from SU1 and SU2 will be combined to form a DU ICS which will then be submitted for laboratory analysis of arsenic, cadmium and lead. The remaining volume of soil in the SU ICS will be archived for potential future analysis including lead speciation.

Should the investigation indicate that a removal action is warranted, it may be of interest to know whether yard soil would need to be handled as hazardous waste should it be excavated. Instructions will be relayed to analytical personnel on selected DU ICS samples that toxicity characteristic leaching procedure (TCLP) analysis will be performed.

4.4 Number of Samples, and Container and Preservation

The estimated number of samples for laboratory analysis is provided in the following table. Note that samples receiving XRF analysis only are not included. The actual number of samples submitted will depend on lead concentrations observed during XRF analysis and visual observations made during collection of the samples.

Matrix	Analytes	Number of Samples
Soil	Lead, Cadmium, Arsenic	60
Soil	PAHs, Toxaphene, PCBs	3
Soil	PDBEs	6
Wipes	Lead, Cadmium, Arsenic	20
Surface Water	Lead, Cadmium, Arsenic, Zinc, Copper, Hardness	10
Sediment	Lead, Cadmium, Arsenic, Zinc, Copper	10

Refer to the following tables for container and preservation requirements on all samples. Note that soil and sediment samples will initially be collected in large resealable plastic bags for transport to ESP. Once samples have been air dried, disaggregated, sieved, and analyzed by XRF, they will be submitted for laboratory analysis. All samples will be collected in certified-clean containers and preserved in the field as appropriate.

Soil/Sediment Samples			
Parameters	**Container(s)/Volume**	**Preservative(s)**	**Holding Time**
Total Metals (As,Cd,Cu,Pb,Zn)	One or more 8-oz glass jars or a 1-gallon resealable baggie	Cool, 2^0C	6 months
PAHs, Toxaphene, PCBs, PBDEs	One or more 8-oz glass jars or a 1-gallon resealable baggie	Cool, 2^0C	6 months

Surface Water Samples			
Parameters	**Container(s)/Volume**	**Preservative(s)**	**Holding Time**
Total & Dissolved Metals/Hardness (As,Ca,Cd,Cu,Pb,Mg, Zn)	One 250ml plastic Nalgene bottle	HNO_3 to pH<2, Cool	6 months
Dissolved Metals	One 250ml plastic Nalgene bottle	Filter (0.45 µm), HNO_3 to pH<2, Cool	6 months

Wipe Samples			
Parameters	**Container(s)/Volume**	**Preservative(s)**	**Holding Time**
Total Metals (As, Cd, Pb)	One 50ml plastic extraction vial	Cool, 2^0C	6 months

4.5 Chain-of-Custody

The ICS soil samples will be stored in the plastic bags in which they were collected. Each bag will be labeled with a unique DU identifier, date, collector initials, and depth using permanent marker. The samples will be recorded on a separate COC form (Appendix B). The samples will remain in the custody of ESP field personnel during sample processing and XRF analysis. Those samples identified for laboratory analysis will be placed into appropriate sample containers and entered onto an ESP COC form to be relinquished to a sample custodian at the state's environmental laboratory for analysis.

5.0 Quality Control

5.1 Field Decontamination

Clean disposable latex gloves will be worn by sampling personnel and clean or field decontaminated equipment will be utilized for each separate DU to minimize the possibility of cross-contamination. Reusable soil sampling equipment will be cleaned between DUs as follows:

- Scraping with putty knife or similar tool to remove soil clumps;
- Brushing with stiff-bristle nylon brush to remove visible soil debris;
- Immersion in a 5-gallon bucket of soapy water and further brushing;

- Rinsing tool with DI water;
- Wiping dry with clean paper towels

5.2 Quality Assurance/Quality Control (QA/QC) Samples

The following samples will be collected as part of the quality control/quality assurance procedures for the investigation.

5.2.1 Equipment Rinsate Blank

An equipment rinsate blank will be collected after decontaminating the soil coring tool between DUs once per day of sampling. Following decontamination of the tool, DI water will be rinsed over the core cylinder and into a sample container which will travel with the other samples back to the laboratory for analysis.

5.2.2 Duplicate Surface Water Sample

A duplicate surface water sample will be collected at the downstream sampling location nearest the facility in accordance with the SOP MDNR-ESP-210.

5.2.3 Duplicate Sediment Sample

A duplicate sediment sample will be collected at the downstream sampling location nearest the facility in accordance with the SOP MDNR-ESP-210.

5.2.4 Replicate Field ICS

Replicate ICS will be collected to measure precision of the overall soil sampling and analysis process, and to provide data for calculating a conservative estimate of the mean concentration. Within 20% of the SUs, after the initial ICS is collected, a total of three ICS will be collected in an identical manner, except the increment locations will be off-set as much as possible from each

within the SU/DU. The replicate samples will be labeled with the DU/SU name and "replicate 1", "replicate 2", and "replicate 3". SUs will be chosen for replicate sampling based on the proximity of the yard to the facility, in-situ XRF results if available, and other field observations. An effort will be made to conduct replicate sampling at residences both near to facility and further away along haul routes. Replicate sampling will also be conducted at 2 of the background residences.

A relative standard deviation less than 30% will generally indicate acceptable precision; however the degree of precision required depends on how close the estimated mean concentration is to the screening level. Thus, more data variability may be tolerable if the concentrations measured are either well above or well below the screening levels.

5.2.5 XRF Precision Samples

The precision of bagged sample XRF analyses will be evaluated by conducting multiple analyses of selected samples at a frequency of 5% for bagged sample analysis and once per day per analyzer for in-situ analysis. For bagged samples, the precision samples will be selected based on lead concentrations. Samples will be chosen to reflect the full range of concentrations observed, however, special emphasis will be placed on selecting samples near the screening levels if possible. The selected sample will be analyzed seven separate times without moving the bagged sample (without moving the analyzer for in-situ analysis) between each analysis. The relative standard deviation among the multiple analyses will be assessed as an indication of instrument precision.

5.2.6 Laboratory Subsampling Replicates

Laboratory subsample replicates will be requested on selected ICS samples submitted to the laboratory. The laboratory will subsample the container two times and conduct two separate analyses. This procedure measures the within-sample container matrix heterogeneity and the error associated with subsampling the container. Samples will be identified for replicate subsampling by indicating in the "comments" field of the ESP COC form.

5.2.7 Laboratory QC

Laboratory precision and accuracy will be assessed as described in the QAPP for Pre-Remedial/Pre-Removal and Targeted Brownfields Site Assessments Revision 6, December 7, 2007, and ongoing.

6.0 Investigation Derived Wastes (IDW) Plan

Efforts will be made to minimize IDW generation. IDW may include soil, sediment, decontamination fluids, disposable sampling equipment, and disposable personal protective equipment (PPE).

Field personnel will attempt to return unused soils to their source immediately after generation or, if warranted, containerize and return to the ESP laboratory for proper disposal. Disposable PPE and disposable sampling equipment will generally be handled as solid waste, containerized, and properly disposed. Wash and rinse waters generated during equipment decontamination will generally be discharged to the ground on-site or, if warranted, containerized and returned to the ESP laboratory for proper disposal.

7.0 Site Safety

A safety briefing will be held on-site prior to initiating field activities and field personnel will be required to read and sign the site-specific health and safety plan. The site safety plan is attached as Appendix A.

8.0 Reporting

ESP will provide a copy of the chain of custodies and laboratory result sheets. SAU will prepare an Investigation Report.

References

Festus, 2011 St. Louis Testing Laboratory, Report of Test Results – Street Sweepings Sample, City of Festus, August 12, 2011.

MDNR, 2007 Missouri Department of Natural Resources, QAPP for Pre-Remedial, Pre-Removal and Targeted Brownfields Assessments, December 7, 2007.

MDNR, 2009 Missouri Department of Health and Senior Services, Lead Poison Prevention Manual, 2009. http://health.mo.gov/living/environment/lead/manual/index.php

MDNR, 2011a Missouri Department of Natural Resources, RCRA Site Investigation Report, September 14, 2011.

MDNR, 2011b Missouri Department of Natural Resources, Report of Compliance Inspection November 23, 2011.

MDNR, 2011c Missouri Department of Natural Resources, RCRA and Missouri Hazardous Waste Management Law Compliance Evaluation Inspection Report, November 22, 2011.

MDNR, 2012 Missouri Department of Natural Resources, Data from January 2012 Sampling Event (not yet published).

NPN, 2011 Letter to Mr. Greg Shapiro, Shapiro Brothers/MW Recycling, from David B. Rowe, NPN Environmental, December 12, 2011.

USEPA, 1990 U.S. Environmental Protection Agency Hazard Ranking System, 40 CFR Part 300, Appendix A, 55 FR 51583, December 14, 1990, http://www.thefederalregister.com/d.p/2007-05-25-E7-10055.

USEPA, 1992 U.S. EPA, Hazard Ranking System Guidance Manual, EPA/540/R-92/026, November, 1992. http://www.epa.gov/superfund/sites/npl/hrsres/index.htm

SIGNATURES

Prepared by:

Michael Stroh
Environmental Specialist
Site Assessment Unit
Hazardous Waste Program

Sean Counihan
Environmental Specialist
Field Services Unit
Environmental Services Program

Approved by:

Julieann Warren
Unit Chief
Site Assessment Unit
Hazardous Waste Program

Date: 3-27-2012

APPENDIX A

Health & Safety Plan

MISSOURI DEPARTMENT OF NATURAL RESOURCES
DIVISION OF ENVIRONMENTAL QUALITY
ENVIRONMENTAL SERVICES PROGRAM

SITE HEALTH AND SAFETY PLAN

1.0 INTRODUCTION

This plan has been prepared for implementation by DNR employees, using operating procedures for which they are specifically trained. Any use of the plan by other agencies, organizations, or private individuals is at their own risk.

2.0 KEY PERSONNEL

MDNR OSC: Sean Counihan SAFETY OFFICER: Sean Counihan

OTHER MDNR PERSONNEL/TITLE:

Enivirnmental Specialists Ken Hannon, Michael Stroh, Valerie Wilder, Shelly, Jackson, Paul Embree, Joe Twellman, Darren Bernat,

3.0 SITE INFORMATION

Site name Shapiro Brothers Scrap Yard County/City: Jefferson County/ Festus

Sampling date: 04/09/2012 Site Description: The site is located at 9th Street and Delmar Street. It is a scrap metal processing plant. They buy and sell scrap, dismantle railroad cars, and are a demolitions contractor. After complaints about dust and dirt covering houses and vehicles, the city collected a composite soil sample from the vicinity of the business at 9th St., Delmar Street, and Vine Street. The results of that sample indicated high levels of lead. Samples from a MDNR RCRA investigation in September of 2011 confirmed the presence of lead in hazardous amounts, whereas samples collected and analyzed by the PRP did not. This additional sampling will help evaluate the site for further action.

3.1 Overall Incident Risk/Hazard Analysis

Chemical: _____Serious _____Moderate X____Low _____Unknown

Physical: _____Serious _____Moderate X____Low _____Unknown

3.2 Contaminant(s) of Concern: **Contents are unknown**

3.2.1 Physical State: _____Liquid X_____Solid _____Sludge _____Gas/Vapor

Chemical Characteristics: (check all that apply)

_____a. carcinogen _____b. biological _____c. corrosive _____d. combustible
_____e. explosive _____f. flammable _____g. volatile X_____h. poison
_____i. radioactive _____j. reactive _____k. other: Unknown Chemicals_____

3.2.2 Physical Hazards: (check all that apply)

a. _____overhead _____b. below grade _____c. confined space __X____d. noise
e.X_____splash _____f. fire/burn X_____g. puncture _____h. heat stress
i. X_____cut X_____j. slip/trip/fall _____k. cold stress _____l. electrical
m. _____mechanical/heavy equipment _____n. other:_____

* The need for confined space entry by ESP personnel shall be evaluated on a site-by-site basis. A confined space entry permit must be signed by the appropriate Unit or Section Chief prior to ESP employees entering a confined space (29 CFR 1910.146). Confined space entry shall be screened in at least Level B prior to downgrade. **Adequate resources must be available and specific planning and tasks determined before confined space entry is initiated.**

3.3 Task-Specific Risk Analysis (attach additional sheets as necessary)

Task Description	Chemical Hazards	Physical Hazards	Level of Protection
Sample collection	h	e d g i j	Level D
	a b c d e f g h i j k	a b c d e f g h i j k l m n	
	a b c d e f g h i j k	a b c d e f g h i j k l m n	
	a b c d e f g h i j k	a b c d e f g h i j k l m n	

4.0 MEDICAL SURVEILLANCE AND PERSONNEL TRAINING REQUIREMENTS

All ESP field personnel participate in a medical monitoring program and are trained at least to the level of "Hazardous Substance Emergency Response-Technician" as required and specified in the department's written health and safety program located in Section 2 of the MDNR-Hazardous Substances Emergency Response Plan (HSERP). The written policy satisfies requirements set out in 29 CFR 1910.120. MDNR ESP's respiratory protection program meets the requirements of 29 CFR 1910.134.

ESP personnel will ascertain as much information as possible regarding health and safety issues associated with the

site prior to initial entry. Information shall include chemical and physical hazards as listed above, types and amounts of materials involved, and citizens/areas threatened by the incident.

5.0 PERSONAL PROTECTIVE EQUIPMENT

ESP shall utilize the Protection Level categories defined in 29 CFR 1910.120, Appendix B, and known as Levels A, B, C, and D. Refer to Section 2 of the MDNR-HSERP for definitions of Protection Levels. ESP personnel shall inspect APRs and SCBAs at least monthly and maintain a record of such to ensure equipment is functional.

Levels of protection shall be reassessed and upgraded as conditions change and information is updated to comply with worker safety while performing site activities.

Action Levels for evacuation of work zone pending reassessment of conditions:

X Level D: O_2 < 19.5% or > 25%; explosive atmosphere > 10% LEL; organic vapors > background levels; othe

X Level C: O_2 < 19.5% or > 25%; explosive atmosphere > 20% LEL; organic vapors (in breathing zone) > 5 m.

X Level B: O_2 Explosive atmosphere > 20% LEL; unknown organic vapors (in breathing zone) > 500 m.u.; othe

X Level A: ESP personnel shall evaluate the need for entry on a site-specific basis and may utilize its emergency response contractor for Level A situations which may arise.

6.0 FREQUENCY AND TYPE OF AIR MONITORING/SAMPLING

Instrument	Contaminant of Concern	Sample Location (Area/Source)	Frequency	Odor Threshold/ Description

7.0 SITE CONTROL MEASURES

7.1 The "Buddy-System": ESP personnel performing any work activities within the exclusion zone shall employ the "buddy-system" at all times, as required and defined in Section 2 of the MDNR-HSERP. The "buddy-system" may not be required while an ESP staff member is observing or providing oversight of cleanup activities performed by a contractor or responsible party.

7.2 Safe work Practices: Refer to Section 2 of the MDNR-HSERP for written safety practices to be followed at all times by ESP personnel while on-site at an incident.

7.3 Site Communications: The use of two-way radios or establishment of hand signals for communications shall be determined prior to entering the work zone and followed by ESP personnel.

7.4 Radiation Safety: Due to the possibility of an unknown radiation hazard being present on a site, ESP personnel shall be required to wear radiation indicator badges (TLD badges) while on-site.

7.5 Work Zones: ESP personnel shall ensure work zones are established and be aware of their locations.

8.0 DECONTAMINATION PROCEDURE/SOLUTIONS:

Personnel: Gloves and clothing will be placed in a garbage bag and returned to Jefferson City for proper disposal.

Equipment: Returned to Jefferson City for proper decontamination.

Instruments: Returned to Jefferson City for proper decontamination or disposed of back in Jefferson City.

Decontamination fluids/materials may be to be containerized for proper disposal.

9.0 EMERGENCY INFORMATION:

In the event of an emergency, notify the MDNR Environmental Emergency Response Office at 573/634-2436. The Duty Officer will make the appropriate notifications.

10.0 ADDITIONAL EMERGENCY INFORMATION/NUMBERS:

Hospital: Jefferson Regional Hospital 636-933-1000

Location/Specific directions from Site: 1400 Highway 61 South, Festus, MO- refer to map

	Name/Location	Telephone Number
Ambulance:	Joachim-Plattin Ambulance 619 Collins Drive, Festus, MO	636-937-2224
Police/Sheriff:	Festus Police Department 100 Park Ave. Festus, MO	636-931-3646
Fire:	Festus Fire Department 212 North Mill Street, Festus, MO	636-937-6646

Poision Control:

Cellular Telephones/Other: Sean Counihan mobile 573-644-3697

1) Central Accident Reporting Office- WORK RELATED INJURY 1-800-624-2354

This number is to be called in the event of a NON LIFE THREATENING injury PROIR to seeking medical care.

11.0 SIGNATURES

ESP personnel shall certify they have read the plan and addressed any questions regarding worker health and safety by signing and dating below followed by printing their name and title.

Signature	Printed Name/Title	Date	TLD Badge

APPENDIX B

Field Sheets:

Chain of Custody Form

Site Sketch Form

Missouri Department of Natural Resources

Team No.____

SOIL SAMPLE LOG
Shapiro Brothers Site, Festus, MO

Loc. ID	Sample ID (SU/DU#)	Date Collected	Time Collected	Collector's Name	Comments

Missouri Department of Natural Resources

SHAPIRO BROTHERS SITE
RESIDENTIAL YARD FIELD SHEET
LOCATION ID: STREET:

N

XRF SerNum(s)_____ Date Screened/Sampled_____

Team Member(s):_____

APPENDIX C

XRF SOP

Standard Operating Procedure for XRF Analyzers
Shapiro Brothers Salvage Yard SI/RSE Investigation - Bagged Samples

In-situ surface soil analyses will be conducted in the field along transects established perpendicular to residential structures. Bagged soil samples will be analyzed with the analyzer in the testing stand and controlled from a laptop PC. A 30-second analysis time will be used for in-situ testing and 45 second analysis time for all bagged samples. *Note, do not operate the laptop PC software with the laptop connected to the network servers.*

All XRF analyses will be recorded either on field sheets or in a written log book for each instrument. The analyst will record the date, the XRF run number (automatically generated by the XRF), sample ID information, and the total Pb result in mg/kg.

I. Startup

- Power up the analyzer and start the InnovX PC software.
- The instrument will automatically perform an initialization procedure, which lasts for 1-2 minutes.
- Following initialization, place the stainless steel standardization disc over the instrument's sampling window in the test stand and close the stand cover.
- Click the "Standardize" button from the upper left window titled "Soil" in the PC software. The instrument will perform an internal 60-second standardization procedure. During standardization, and any other time the x-ray tube is on, the red light on top of the test stand will flash. When the x-ray tube is off, the red light will remain on solid. ***Do not open the test stand lid when the light is flashing.***
- Following standardization, an information window will pop up displaying the analyzer resolution. Record the resolution in the XRF Log Book along with the Run number automatically assigned by the analyzer.
- The analyzer is now ready to analyze standard reference materials (SRMs).

II. Calibration Check

- The NIST 5861 (41ppm Pb), 4315 (244ppm), 2586 (431ppm), 2711 (1162), and 2710 (5532ppm), and Blank SRMs will be analyzed at the beginning of each use. Selected SRMs will also be reanalyzed periodically during the day based on site findings and length of time in the field.
- On the PC software main menu bar, select "Edit" and then chose "Edit Test Information". A data entry window will pop up allowing input of information about the next test.
- Select your name from the "Analyst" dropdown menu & select the check sample from the "Chk_Sampl" dropdown list.

- Click the "Start" button in the Soil Window in the upper left corner of the screen to initiate the test.
- Assess instrument calibration by comparing the measured values to the control chart prepared for each analyzer for each SRM. Verify that the result is within 2SD of the control chart mean, if so, continue to In-situ or Bagged Sample Analysis sections below.
- If values outside 2SD of control chart mean are observed, re-analyze the calibration check sample, or if there are more than one SRM sample cup, analyze another one. If the measured value is still outside 2SD, re-standardize as described above, and re-analyze the standard(s). If the result is still outside 2SD, note the failure in the logbook. If more than one check sample fails this procedure on a given day (particularly if the exceedances are in the same direction – e.g. both 2SD above or below the mean), it may be necessary to update the control charts, manually enter new calibration response factors into the analyzers, or both.

III. Sample Analysis

All XRF analyses will be conducted using InnovX X-ray tube analyzers. In the event of instrument malfunction in the field, in-situ analyses may also be conducted using Niton Xli700 Amerecium^{232} isotope analyzers.

In-Situ Analysis

- Following successful calibration check, click Edit from the main menu bar and select Edit sample Information.
- Enter all applicable information about the first in-situ analysis into the iPAQ using the dropdown and direct edit fields.
- Remove any vegetation from the soil surface exposing an area of approximately five square inches. Shake out any soil clinging to vegetation roots back onto the cleared spot and use a stainless steel spoon or other tool to prepare a one square inch area on which to place the XRF analyzer window. Place a thin-walled plastic baggie over the analyzer snout to protect it from the soil.
- Initiate a 30 second analysis. The analyzer can be operated hands-free using the tripod attachment.
- Record the information in columns on the XRF Data Field Sheet, and continue with additional analyses.
- When data from a given distance for a transect is collected, calculate and record the average on the field sheet.
- Periodically and at the end of the day, re-analyze the SRMs as described in the Calibration Check section above.
- Once per day per analyzer, conduct a precision check by repeating an in-situ analysis 7 times without moving the analyzer between tests. The precision check should be conducted in an area found to contain elevated lead, preferably a concentration near the 400 mg/kg action level.

Bagged Sample Analysis (InnovX analyzers)

- Following successful calibration check, click Edit from the main menu bar and select Edit Sample Information.
- Enter all applicable information about the first sample to be analyzed from the bag label, using the dropdown menus and direct edit fields
- Gently roll the sieved soil around inside the bag to homogenize;
- Place the sample over the analyzer's sampling window ensuring that the soil and bag are in as close contact with the window as possible.
- Close the stand cover.
- Click the Start button from the Soil window to initiate the test.
- The data being acquired will appear in the Chemistry window in the lower center of the PC screen during analysis.
- After analysis, the results will appear in the Results window on the PC.
- A running list of the analyses will appear in the window at the lower left of the PC screen.
- The sample information will remain from the previous test, so no changes are necessary for subsequent replicate analyses on a given sample bag.
- Roll the sieved soil around inside the bag, and re-analyze. Repeat analysis 2 more times.
- Calculate a mean and 95%UCL for the replicate analyses.

> If the mean and the 95% UCL lead concentrations straddle the 400ppm or 1,200 ppm screening level, conduct 3 additional replicate analyses, recalculate the mean and UCL and reassess. This process may need to be repeated for another round of 3 analyses in an effort to get both the mean and UCL on one side of the screening level. It may not be possible in all cases to do this (e.g. when the mean and UCL are very close to the screening level). If after 10 replicate analyses, the mean and UCL still straddle the screening level, the sample will be considered to contain lead above the screening level

- After completing replicate analysis on a bagged sample, click the Edit Sample Information again and enter information for the next bagged sample as above.
- Place the second sample in the test stand, close the cover and initiate the analysis.
- Repeat for remaining samples
- An instrument precision check will be conducted at a frequency of 5%. This will consist of analyzing a sample seven separate times <u>without</u> moving the sample in between each analysis. The %RSD on the replicate analyses should not exceed 15%. Select a bagged sample for the precision check that has elevated lead levels, preferably a concentration near the action level of 400 mg/kg if possible.

IV. Data Downloading

- After the last analysis for the day, select Readings from the main menu bar, and chose Export Readings.

- In the Export pop up box, verify that the "Export readings on date" radio button is selected, the Mode to export is "All", and today's date is circled on the calendar.
- Click OK.
- Insert a USB thumb drive in the laptop, download data to it, and then move data onto network server. Select the directory and file name for the downloaded data. For this project, file naming convention is date & XRF serial number (e.g. 10_14_11_5434)
- Verify that the file type is "Comma Separated Values", and click Save.
- A message will pop up indicating a successful download, and asking whether you would like to open the file. Select Yes, and file will open in Excel. Verify that the data appears correct. Make any corrections you had noted in the run log book.
- Choose Save As from the File menu, and select File Type "Microsoft Excel 97 Workbook.
- Close the InnovX software, power down the analyzer, and shut down the laptop PC.
- Copy the file from thumb drive to the network as soon as possible after analyses. Files will be stored in the H:/Sections/Superfund/SiteFiles/Shapiro/XRF data directory.

Note: For any operation that requests a password, the administrator password is lower case z, and the factory password is 1234.