

1 **FIELD SAMPLING PLAN AND GENERAL QUALITY ASSURANCE**

2 **PROJECT PLAN FOR A**

3 **PILOT STUDY TO ASSESS VOLUME OF MINE WASTE AND**  
4 **CONCENTRATION OF SELECTED METALS IN STREAM AND**  
5 **FLOODPLAIN SEDIMENTS WITHIN THE TRI-STATE MINING**  
6 **DISTRICT IN KANSAS, MISSOURI, AND OKLAHOMA**

7 **PREPARED FOR**

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9 **U.S. Fish and Wildlife Service**

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1 **Contents**

2 **TABLES..... 3**

3 **1.0 INTRODUCTION ..... 4**

4 1.1 PROBLEM ..... 5

5 1.2 DESCRIPTION OF PROJECT OBJECTIVES AND SCOPE ..... 5

6 1.3 PROJECT ORGANIZATION..... 7

7 **2.0 APPROACH AND METHODS ..... 11**

8 2.1 STREAM REACH SELECTION..... 11

9 2.1.1 Identification of Candidate stream reaches ..... 11

10 2.1.2 Field Reconnaissance..... 11

11 2.1.3 Study reach selection and initial GIS mapping ..... 12

12 2.2 SAMPLE SITE SELECTION AND METHODS ..... 12

13 2.2.1 Task 1. In-channel core site selection ..... 13

14 2.2.2. Task 2 Flood-plain core site selection ..... 16

15 2.2.3 Task 3 Determination of streambed sediment depth ..... 17

16 2.2.4 Field GPS survey..... 17

17 2.3 SAMPLE HANDLING AND FIELD PROCESSING ..... 18

18 2.3.1 Sample collection ..... 18

19 2.3.2 Other sediment sample collection methods ..... 19

20 2.4 BOREHOLE GEOPHYSICS ..... 22

21 2.5 SAMPLE HANDLING AND PROCESSING..... 22

22 2.6 ANALYTICAL METHODS..... 24

23 2.6.1 Metal screening by X-Ray Fluorescence (XRF)..... 24

24 2.6.2 Laboratory Analyses ..... 25

25 2.7 QUALITY ISSUES..... 27

26 2.7.1 Quality Control ..... 27

27 2.7.2 Data Quality Objectives for Measurement Data..... 28

28 2.8 DOCUMENTATION ..... 30

29 2.9 GEOSPATIAL ANALYSIS ..... 32

30 **3.0 DELIVERABLES AND SCHEDULE ..... 32**

1 3.1 DELIVERABLES..... 32

2 3.2 PROJECT SCHEDULE ..... 33

3 **4.0 REFERENCES ..... 34**

4

5

6 **List of Figures (at the back of the report)**

- 7 **Figure 1.** Location of candidate stream reaches to be investigated in the Tri-state Mining District
- 8 (TSMD).
- 9 **Figure 2.** Theoretical layout of in-channel transect and floodplain core sample sites along a
- 10 candidate representative study reach along Center Creek
- 11 **Figure 3.** Theoretical layout of in-channel bias and floodplain core sample sites along a candidate
- 12 representative study reach along Center Creek
- 13 **Figure 4.** Generalized X-Ray fluorescence (XRF) screening approach for a sediment core.

14

15 **Tables**

- 16 **Table 1.** Summary of key project team members..... 7
- 17 **Table 2.** List of target elements to be determined by Inductively-Coupled Plasma Atomic
- 18 Emission Spectroscopy (ICP-AES) in laboratory samples. .... 26
- 19 **Table 3.** Generalized project timeline. .... 33

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2           **Field Sampling Plan for a Pilot Study to Assess the**  
3 **Volume of Mine Wastes and Concentration of Selected**  
4 **Metals in Stream and Floodplain Sediments within the**  
5 **Tri-State Mining District in Kansas, Missouri, and**  
6 **Oklahoma**

7

8 **1.0 Introduction**

9 Lead and zinc mining beginning in the mid-1800's in the Tri-State Mining District (TSMD) has  
10 left a legacy of mine waste distributed throughout the region. Waste products from the mining,  
11 milling and extraction of ore include chat and tailings which contain elevated concentrations of  
12 lead (Pb), zinc (Zn), and cadmium (Cd). Generally, chat is small gravel size material (0.6 – 1.6  
13 cm) while tailings are sand sized (2 mm or less). Because of the size of tailings, they are easily  
14 mobilized into streams and waterways and transported great distances from their source. Over  
15 time, chat and tailings have become incorporated into streams resulting in increased  
16 concentrations of Pb, Zn, and Cd in sediments. Because of the easily mobilized nature of tailings,  
17 during flood events, tailings may also be deposited in overbank and floodplain deposits and could  
18 be transported downstream when these sediments become reintroduced into the active stream  
19 channel. The environmental contamination caused by decades of mining activity resulted in  
20 several counties in the region being listed on the U.S. Environmental Protection Agency's  
21 (USEPA) National Priority List as Superfund hazardous waste sites.

22

23 Currently (2010) the USEPA is actively remediating mine sites (tailings and coarser "chat"  
24 dumps, mine shafts, mine seeps, and associated facilities) in the TSMD. Recent studies by  
25 the U.S. Geological Survey (USGS), in cooperation with the U.S. Fish and Wildlife Service  
26 (USFWS), and the Kansas Department of Health and Environment (KDHE) and USEPA  
27 (Regions 6 and 7) documented Pb, Zn, and Cd concentrations in sediment that far exceeded  
28 background levels as well as probable effects guidelines for adverse biological effects for aquatic  
29 biota (Pope, 2005; Juracek, 2006; Ingersoll and others, 2009; McDonald and others, 2000). These

1 studies sampled deposited sediment in the Spring River and its tributaries Empire Lake in  
2 Cherokee County, Kansas, Tar Creek, and Empire Lake in Cherokee County, Kansas and Grand  
3 Lake O' the Cherokees (Grand Lake) in Oklahoma. The adverse effect of the mining-related  
4 contamination on freshwater mussels was documented by Angelo and others (2007).

## 6 **1.1 Problem**

7  
8 As remediation of mine source areas continues, increased attention is being focused on the extent  
9 and magnitude of metals contamination in area streams, in particular, obtaining reliable  
10 information on the distribution and volume of mine tailings and their associated metals contained  
11 within stream and floodplain sediments in the TSMD. Standard practice for determining the  
12 extent of metal contamination in streambed sediments is the collection of samples from the upper  
13 few centimeters of the streambed, either in a biased manner by targeting depositional areas of  
14 finer sediments behind rocks, etc., or collecting a composite of subsamples along one or more  
15 transects across the stream. While these methods are effective at characterizing the spatial or  
16 downstream extent of contamination, they do not provide information on the depth of  
17 contamination or variability in contamination within stream features, such as bars, riffles, pools,  
18 etc. While some tailings deposits currently are exposed in sand or gravel bars within and adjacent  
19 to the stream, an unknown volume has been transported and redeposited and may be covered by  
20 native sediments or vegetation, or exist in deep pools. The thickness of these deposits is unknown  
21 but likely variable across and along the stream. Presently (2010), while the aerial extent of  
22 contamination is generally known, there is insufficient information to allow for estimates of the  
23 actual volume of contaminated sediments and mass of mining-related metals in the streams or on  
24 the adjacent floodplains.

## 26 **1.2 Description of Project Objectives and Scope**

27  
28 The primary objective of this investigation is to characterize the lateral and vertical extent of  
29 mining-related metals contamination in streambed and adjacent floodplain sediments along  
30 selected representative reaches of streams in the TSMD. This pilot study is designed to  
31 supplement existing surficial streambed sediment data, and an ongoing USEPA floodplain study  
32 in the Cherokee County, Kansas, by providing previously unknown information on the depth of  
33 mine waste contamination within the stream channel (bank to bank) and adjacent floodplain, and

1 allow estimates to be made of the volume of contaminated sediment within the selected stream  
2 reaches. Sediment samples will be collected from within the active stream channel (hereinafter  
3 referred to as in-channel) and from the adjacent floodplain using a variety of techniques and  
4 drilling platforms. An important result of this effort will be a determination of which methods are  
5 most appropriate to obtain sediment samples.

6  
7 A traditional method to characterize subsurface contamination is to obtain depth dependent  
8 sediment samples. Cores of sediment provide an intact representative sample of the sediment/soil  
9 profile at the location of the core and an appropriate density of cores is needed to adequately  
10 characterize the subsurface. However, a traditional sediment-core characterization of affected  
11 streams in the district is impracticable because the heterogeneous nature of the sediments and the  
12 broad scale of the impacted area necessitate a large number of cores and corresponding expense.  
13 A pilot-scale assessment of representative reaches of streams in the Ozark Plateaus and Osage  
14 Plains physiographic provinces in the TSMD is proposed. Results from this pilot assessment will  
15 provide information on the vertical and lateral extent of mine waste and associated metals and  
16 allow for comparison of several methods to estimate volumes of contaminated sediments along  
17 the study reaches. This information, combined with geospatial information, could then be used to  
18 extrapolate to the full length of the streams draining the TSMD or design a cost-efficient method  
19 for a district-wide assessment of the volume of mine tailings and mass of metal contamination in  
20 streams across the TSMD. Data density within these representative reaches will be higher than  
21 anticipated during a district-wide implementation.

22  
23  
24 Preliminary discussion with the USFWS and USEPA, have defined streams of interest for this  
25 study. Sediment samples will be collected from representative reaches on Center Creek, Turkey  
26 Creek, and Shoal Creek in Missouri, Tar Creek in Oklahoma, and along two reaches of the Spring  
27 River in Kansas. Up to 25 samples will be collected from each selected reach and the adjacent  
28 flood plain. Core samples will be logged by a field geologist and screened with X-ray  
29 fluorescence (XRF) for bulk concentrations of Pb, Zn, and Cd. A subset of the field XRF scans  
30 will be verified with laboratory split samples. A geomorphic analysis of the study reach will be  
31 done to provide an estimate of the volume of unconsolidated sediments within the stream channel  
32 and fraction of those sediments contaminated with mine waste utilizing a Geographic Information  
33 System (GIS) and field estimates of sediment thickness using tile probe measurements and a field  
34 differential Global Position System (DGPS) survey of primary channel features (primarily bars).

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Data will be collected at several scales, such as digitizing features using the 2009 imagery and the GIS versus field DGPS mapping of primary channel features and the comparison of higher density sediment or composite sediment samples to lower density sampling methods, to provide information for optimization of methods that might be used for scaling up to a larger district-wide assessment. Results of investigation, including the evaluation of sediment collection methods, geomorphic analyses, and analytical data will be published in a USGS interpretive report.

**1.3 Project organization**

The following is a summary of project team members, organizational structure, and responsibilities. The project is a coordinated effort between the USGS Missouri and Oklahoma Water Science Centers (WSC). The USGS will organize two field teams to meet the two general project tasks of in-channel and floodplain core sampling (Table 1). The in-channel sampling team will be headed by the Oklahoma WSC and the floodplain coring team will be headed by the Missouri WSC. Except for Tar Creek, the Missouri WSC is primarily responsible for the reconnaissance, selection of the study reaches, obtaining landowner consent and utility clearances, conducting floodplain sampling, and maintaining the project GIS database. The Missouri WSC also will be responsible for coordination with the ongoing USEPA Spring River Floodplain study in Cherokee County, Kansas and ensuring data compatibility between the two studies. Data review and compilation, database management, and GIS support is primarily the responsibility of the Missouri WSC. The Oklahoma Center is primarily responsible for initial preparation of the SAP, in-channel coring activities, and will lead all activities at the Tar Creek study area. The Oklahoma WSC will also be responsible for coordination of XRF analytical support from the USFWS, as needed. The final report will be jointly co-authored by both the Missouri and Oklahoma WSC. Key individuals participating in the project and their specific roles and responsibilities are discussed below:

28 **Table 1.** Summary of key project team members.

| <b>Function</b>         | <b>Team member</b>       | <b>Project Role</b>         | <b>Title</b>                        | <b>Office</b> |
|-------------------------|--------------------------|-----------------------------|-------------------------------------|---------------|
| Project Management Team | Jim Dwyer                | Overall project coordinator | Environmental Protection Specialist | USFWS         |
|                         | Dave Mosby (or designee) | USFWS Analytical Support    | Fish and Wildlife Specialist        | USFWS         |

|                          |                  |                                |                         |              |
|--------------------------|------------------|--------------------------------|-------------------------|--------------|
|                          | Mike Slifer      | USGS Approval official         | Director                | Missouri WSC |
|                          | John Schumacher  | USGS/FWS project liaison       | Supervisory Hydrologist | Missouri WSC |
|                          | Doug Mugel       | Project principle investigator | Hydrologist             | Missouri WSC |
| In-channel field team    | Mark Becker      | Team leader                    | Hydrologist             | Oklahoma WSC |
|                          | --               | Team member                    | Technician              | Oklahoma WSC |
|                          | Jacob Morris     | Team member                    | Technician              | Missouri WSC |
|                          | Mike Kleeschulte | Team member                    | Hydrologist             | Missouri WSC |
|                          | Doug Mugel       | Team member                    | Hydrologist             | Missouri WSC |
| Floodplain sampling team | Doug Mugel       | Team leader                    | Hydrologist             | Missouri WSC |
|                          | Paul Brenden     | Team member                    | Technician              | Missouri WSC |
|                          | Jacob Morris     | Team member                    | Technician              | Missouri WSC |
|                          | Mike Kleeschulte | Team member                    | Hydrologist             | Missouri WSC |
|                          | Mark Becker      | Team member                    | Hydrologist             | Oklahoma WSC |
| Project GIS              | Joe Richards     | GIS specialist                 | Hydrologist             | Missouri WSC |
| Project Reporting        | Mark Becker      | Project lead author            | Hydrologist             | Oklahoma WSC |
|                          | Doug Mugel       | Project coauthor               | Hydrologist             | Missouri WSC |
|                          | John Schumacher  | Project coauthor               | Hydrologist             | Missouri WSC |
|                          |                  |                                |                         |              |

1

2 **Jim Dwyer, USFWS, Project Manager** – the primary decision maker for the project and the  
3 primary user of the data to determine if project results meet the study objectives and if further  
4 investigation in the study area is required and to what extent. His primary duties are:

- 5 - Overall responsibility for the investigation.
- 6 - Reviewing and approving the project SAP.
- 7 - Reviewing reports and ensuring plans are implemented according to schedule.
- 8 - Coordinate with Tri-State Case Managers related to study plans, implementation, results,  
9 and reporting.

10

11 **Dave Mosby (or designee) USFWS Fish and Wildlife Biologist**

- 12 - Provide XRF analytical support in the field and/or laboratory

13

14 **Mike Slifer, USGS Approving official** –Director of the Missouri Water Science Center; will  
15 serve as the approving official for all USGS activities for the project. Specific project duties  
16 include:

17

- 17 - Reviewing and approving the project SAP.
- 18 - Ensuring that study objectives are relevant and within the mission of the USGS.
- 19 - Reviewing reports and ensuring plans are implemented according to schedule.



- 1 - Making final project decisions with the authority to commit the necessary resources to
- 2 conduct the project.
- 3 - MOWSC approving official for the project final report.

4

5 **John Schumacher, Overall USGS Project coordinator** – The USGS Project coordinator has  
6 the following specific responsibilities:

- 7 - In conjunction with project technical team, revises the SAP as needed to define project
- 8 goals and activities.
- 9 - Serve as a liaison between USGS and FWS and coordinates overall field and laboratory
- 10 activities (field XRF screening) associated with the project and prepares project updates.
- 11 - Coordinates with project team members to ensure project operates within allotted schedule
- 12 and budget.
- 13 -Provides technical assistance to the project team members on matters related to geochemical
- 14 processes, sampling protocols, and quality-assurance (QA) issues.
- 15 - Coordinates with ongoing USEPA Region 7 studies and activities in the TSMD.

16 **Doug Mugel, Principal Investigator**–Missouri Project Leader. Will oversee project activities in  
17 Missouri, including Center Creek, Turkey Creek, and Shoal Creek. Doug has the following  
18 specific responsibilities:

- 19 - Responsible for reconnaissance and selection of study area on Center, Turkey, and
- 20 Shoal Creeks. This includes coordination and obtaining access from landowners, and
- 21 utility clearances.
- 22 - Serve as head of the floodplain sampling team. Coordinate overall field and
- 23 laboratory activities associated with sampling of flood-plain soil.
- 24 - Conducts project activities in accordance with the SAP within the allotted time frame
- 25 and document and obtains approval for deviations.
- 26 - Verify and validate field and laboratory data generated from sampling activities.
- 27 - Ensures that all relevant project data and sample sites are properly entered into the
- 28 USGS National Water Information System (NWIS).
- 29 - Assists in the preparation of report describing the extent and magnitude of mining
- 30 contaminated sediments in the representative stream reaches studied.

31

32 **Mike Kleeschulte, Project team member**–Missouri. Mike will serve as a field team member  
33 and will provide support to the Principle Investigator and be capable of fulfilling any of the  
34 responsibilities of the Principle Investigator.

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**Mark Becker, Lead Author** –Oklahoma Project Leader. Mark’s specific project duties include:

- Responsible all field activities at the Tar Creek study reach ( reconnaissance, access, utility clearance, sampling).
- Serve as head of the in-channel sampling team. Coordinates overall field activities associated with in-channel sediment sampling.
- Project lead author responsible for preparation of the final study report.
- Verify and validate field and laboratory data generated from sampling activities.
- Ensures that all relevant project data for the Tar Creek area are properly entered into the NWIS.
- Ensures that field activities follow USGS safety protocols and boat operations plans.
- Coordinates project activities with USEPA Region 6 activities in the Oklahoma part of the TSMD.

**Paul Brenden, Hydrologic technician** – Missouri. Drill rig operator. Primary responsibility includes safe operation, maintenance, and cleaning of drill rigs and sampling equipment.

**Jacob Morris, Hydrologic technician** – Missouri. Jacob will function in a key role in the collection of field data and preparation of geologic logs, and compilation and review of field XRF, laboratory, and geologic data. He will also assist with or operate drilling and sampling equipment.

**David Detra, USGS Central Region Mineral Resource Team** –As the coordinator for analytical services by the USGS Central Region Mineral Resource Assessment Team (CRMR), is responsible for coordinating the analysis of sediment samples for laboratory grain-size and elemental analysis and validation of laboratory data. This coordination will include the receipt of samples at the laboratory, selection of the analytical team, verification that internal laboratory audits are conducted per standard CRMR operating procedures (SOP’s). Dave will coordinate internal laboratory QA procedures to include the analysis of standard reference samples (control samples) and split-replicate samples. Associated laboratory QA data and any identified laboratory problems will be reported to the project Principal Investigator as soon as they are determined.

## 2.0 Approach and Methods

Some stream sampling locations will present access problems and difficulties in obtaining representative samples. Initially, data collection will take place at 1 or 2 streams to determine which approach and sampling methods are best suited for achieving the objectives of this project. While it is understood that each stream has its unique attributes that may require a unique approach and different methods, the lessons learned from the initial sampling will be applied to the remaining streams.

### 2.1 Stream Reach Selection

#### 2.1.1 Identification of Candidate stream reaches

Based on discussions with the USFWS and other members of the Trustee Councils of Tri-State, five streams in the TSMD were selected to be included in this pilot study (1) Center Creek, (2) Turkey Creek, (3) Shoal Creek, (4) Tar Creek, and (5) Spring River. One representative study reach will be investigated along each stream except for the Spring River, where two reaches will be selected to coincide with locations of the USEPA Spring River floodplain study (fig. 1). In-channel samples will be collected from all reaches. Samples will be collected from the floodplain of all streams except Spring River, as floodplain coring of Spring River is part of the USEPA Cherokee County study. Stream reaches selected for sampling will be selected based upon the proximity to downstream tributaries, access, and evidence of extensive stream alteration since mining has occurred. Historical aerial photos will be compared with recent photos to determine anthropogenic or natural changes that may affect or bias the results. Stream segments in Missouri selected for sampling must contain several of the geomorphic channel units (GCU) such as pools, riffles, and runs. These are common characteristics in Ozark streams within the TSMD. Tar Creek is a typical prairie stream and does not naturally contain the coarse sediment found in Ozark streams.

#### 2.1.2 Field Reconnaissance

After candidate reaches have been selected a field reconnaissance of the locations will be done to verify that the reaches contain the primary GCUs and that there is adequate access to the stream reach. Notes will be taken on approximate stream width, type and number of physical features within the channel (bars, riffles, manmade features, bank heights, obvious utilities, etc.) and floodplain (natural levees, terraces, old meanders, and cultural features), and a determination made if the reach is wadeable or non-wadeable. Physical conditions of the stream bank (height, slope, vegetative cover, etc.), water depth, and access will dictate the types of equipment that can

1 be used at the site (hand operated, tractor or ATV, vehicle access). During the reconnaissance, a  
2 tentative layout of in-channel and floodplain core locations will be made to estimate the number  
3 of cores and samples to be collected at each candidate reach. A hand-held GPS will be utilized to  
4 provide estimated locations of transects and core locations and to locate noteworthy features  
5 observed such as tailings exposures, utilities, target areas of bias sampling (old cutoff meanders,  
6 etc.) It is anticipated that most access will be on or across private property and an important focus  
7 of the field reconnaissance will be contacting and securing access from property owners. Study  
8 reaches will be moved or adjusted depending on access restrictions. More than one  
9 reconnaissance may be required for each reach.

### 11 2.1.3 Study reach selection and initial GIS mapping

12 Based on the field reconnaissance, study reaches will be selected, a layout of proposed in-channel  
13 sample sites will be done using a GIS. Any specific points of interest noted during the field  
14 reconnaissance (old channel meanders or mine shafts, tailings exposures) that may affect the  
15 study design will be imported into the GIS. The layout will include determination of the length of  
16 the study reach, proposed locations and number of in-channel and floodplain sample locations.  
17 The base map for the study will be 2009 NAIP imagery with overlays of the 1938 Soil  
18 Conservation Service (SCS) imagery. The location of depositional features in the channel (bars  
19 and riffles) observed on the 2009 imagery will be digitized using the GIS to provide an estimate  
20 of the area of these features along each study reach for comparison to the mapping of these  
21 features during the field effort. The results of this comparison will be used to determine the  
22 validity of using GIS mapping to extrapolate beyond the selected reaches to other portions of the  
23 streams. Coordinates of the study reach and of proposed subsurface activities will be used to  
24 obtain the required utility clearances (e.g. Missouri One-Call). More than one visit to each study  
25 reach may be required to stake locations of floodplain core sites and verify locations of marked  
26 utility crossings.

## 28 2.2 Sample Site Selection and Methods

29  
30 The following sections describe the theoretical approach for the collection of samples along each  
31 representative stream reach. One of two general approaches will be used at each stream reach. In  
32 addition to floodplain core sampling, the initial approach for each stream reach will be to collect  
33 in-channel samples at sites along transects. However, because of the expected heterogeneous

1 nature of sediment distribution in at some if not all in-channel streams reaches, the possibility that  
2 regularly-spaced samples along a transect might not adequately represent the sediments in the  
3 reach, and other issues such as access at transects, the transect approach may not yield suitable  
4 samples, and a directed (bias) approach to sites where sediment accumulations are observed may  
5 instead be used. The determination of sample approach will be made during sampling depending  
6 on conditions encountered. Results from initial sampling will help direct the approach in  
7 subsequent sampling. One outcome of this pilot study will be the determination of which of these  
8 approaches is most suitable for sediment sampling of the TSMD. Also, because of the uncertainty  
9 of subsurface conditions and physical limitations regarding the collection of in-channel core  
10 samples, sample methods, including modifications to methods described herein or new methods  
11 will to some extent be determined at the time of sample collection.

12  
13 Sample collection along each representative stream reach will be divided into two tasks. Task 1  
14 will involve the collection of in-channel samples (between top of banks). Task 2 will involve the  
15 collection of core samples from the floodplain adjacent to the sampled stream reach. A third, non-  
16 sample task (task 3) will be to estimate the depth of in-channel sediments along the study reach  
17 using a tile probe or maximum refusal depth from coring. The following describes the general  
18 approach to the layout of sample sites along a representative stream reach. As an example, a  
19 possible layout of in-channel and floodplain cores along a candidate study reach along Center  
20 Creek is shown in figure 2. The candidate reach shown in figures 2 and 3 is along the lower part  
21 of Center Creek about 0.7-mi (miles) upstream from the mouth. This area was identified because  
22 of a complex series of old channel meanders and channel alternations, including a low-water dam  
23 evident on the 1938 air photograph.

24

25 **Figure 2.** Theoretical layout of an in-channel transect and floodplain core sample sites along a  
26 candidate representative study reach along Center Creek.

27

28 2.2.1 Task 1. In-channel core site selection

29

1 A maximum of 20 in-channel core samples will be collected along each representative stream  
2 reach. For the transect approach, in-channel sediment samples are proposed to be collected using  
3 three strategies:

4  
5 Transect sampling --by selecting transects perpendicular to the stream channel crossing the  
6 stream at a specific GCU within the study reach (9 to 15 samples),

7 Longitudinal sampling --by evenly dividing the length of the study reach and placing one  
8 core in the middle of the channel at each interval (3 to 5 samples),

9 Special sampling to target unusual features may be done, to supplement other samples (0 to 2  
10 samples).

11  
12 The number of transects in each GCU (one or two) and spacing of sample sites along each  
13 transect will be dependent on the channel complexity along the study reach and width (between  
14 top of banks) of the channel, and access from adjacent landowners. The spacing of cores along  
15 each transect will be determined by dividing the channel width at that transect location ( $W$ ) by  
16  $(n+1)$  where  $n$  is the number of cores (typically 3 or 5 depending on whether one or two transects  
17 per GCU are done) to be collected along the transect. The first core sample will be collected at  
18  $W/(n+1)$  feet from the top of the right bank (looking downstream). Using this method, core  
19 spacing along each transect will vary depending upon the channel width at each cross section.  
20 Transects will be labeled with a letter designation beginning with "A" and increasing from  
21 upstream to downstream order. Core sites will be numbered from the right bank (looking  
22 downstream) beginning with "01". Generally cores along a transect will not be spaced closer  
23 than about 25 ft, such that only three cores are planned if the channel is less than about 100-ft  
24 wide.

25  
26 As part of the transect approach and to test this approach, longitudinal sampling will be done to  
27 compare with the results from transect sampling. Generally these samples will be collected after  
28 the transect samples have been collected. Spacing of the samples will be done by dividing the  
29 length of the study reach by  $(n+1)$  where  $n$  is the number of longitudinal samples to be collected.  
30 Depending upon the complexity of the channel and number of transect samples collected, three to  
31 five longitudinal samples will be collected. The samples will be collected from the center of the  
32 channel (midpoint between banks) and thus could be from within a pool, riffle, run, or gravel bar,  
33 depending on the feature present at that location. Longitudinal cores will be given a "L" prefix  
34 and numbered in downstream order beginning with "L01" at the upstream location. It is possible

1 that unusual features may be encountered along a study reach that were not sampled by the  
2 transect or longitudinal samples. These features may be selected for special sampling (maximum  
3 of two samples). Such unusual features may include a depositional area of fine grained material  
4 or an accumulation of visually identified tailings immediately upstream or downstream from a  
5 pipeline crossing or low-water bridge. Special samples will be given an “S” prefix and numbered  
6 sequentially in the order collected.

7  
8 The alternative approach to the transect approach is directed (bias) sampling at locations where  
9 sediments are known to occur in substantial thicknesses. This approach may be superior to the  
10 transect approach if the transect sample sites, which are determined by regular spacing along the  
11 transect, are not located where sediments are thick enough to yield samples representative of the  
12 reach, or are located where access or other issues present a problem. If this appears to be the case  
13 at the time of sampling, the decision will be made to collect samples using the bias approach. Up  
14 to 20 samples would be collected along the length of the reach, primarily in bars where sediments  
15 are thick, but also including up to 5 samples in the wetted channel (in-stream) where sediment  
16 character is likely to be different. A hypothetical layout of bias sample sites is shown in figure 3.

17  
18 **Figure 3.** Theoretical layout of in-channel bias and floodplain core sample sites along a  
19 candidate representative study reach along Center Creek.

20  
21 A variety of methods will be used to collect in-channel samples ranging from hand operated  
22 vibrating core and auger to machine driven direct push and split spoon, to hand shoveling , etc.  
23 Collection of core samples is the preferred method of in-channel sample collection. However, it is  
24 anticipated that because of the difficulties in sampling gravel with traditional coring devices,  
25 alternative sampling methodologies are likely to be employed which may or may not result in  
26 actual core samples. The target depth of in-channel samples is variable and dependent upon the  
27 method used to obtain the sample, equipment used, and grain-size range of the material. A target  
28 depth of 3-5 ft beneath the land surface or water-sediment interface is proposed because this is the  
29 maximum practical depth thought to be possible with hand operated devices. However, attempts  
30 will be made to collect samples from depths up to 8 ft or refusal.

1 A hierarchy of sampling methods will be used for in-channel sampling, starting with mechanical  
2 core drill rigs (truck, tractor, or ATV mounted). Locations that are accessible by these drill rigs  
3 (gravel bars and shallow riffles, if access to these locations across floodplains is possible) are  
4 areas where continuous cores to depths to 8 ft may be possible given favorable grain-size and  
5 other conditions, resulting in high-resolution vertical characterization of the sediments and their  
6 metal contents. Where larger cobbles are encountered, those zones may be bypassed or larger  
7 diameter “split spoon” type samples attempted. If grain-size is not too large, the most reliable  
8 samples can be obtained using 2-in diameter core tubes with disposable liners that can be  
9 advanced to specific depths, opened, and “pushed” to obtain core samples from saturated  
10 materials that would otherwise collapse within an unlined borehole. Larger diameter (5-in. or  
11 larger) split spoon samples can be used, however, these larger tools are limited to about 5 ft  
12 below the water table or water-sediment interface because they will be advanced in an open  
13 borehole. Remote areas not accessible by the truck or ATV drill rigs will be limited to hand  
14 operated power equipment. On gravel bars and in shallow (less than about 2 ft of water depth)  
15 riffles several methods ranging from vibratory and gasoline powered augers combined with  
16 vibratory techniques [limited to smaller diameter (2-in or less) core tools] can be attempted (Box  
17 and others, 2001). Hand methods likely will be limited to depths of less than 5 ft. For in-stream  
18 samples in non-wadeable conditions, a floating platform will need to be used. This may consist of  
19 a john boat, dock floats, or a pontoon boat.

20

### 21 2.2.2. Task 2 Flood-plain core site selection

22 Core samples from the floodplain will be collected along each representative reach where in-  
23 channel samples are collected. About 5 to 10 floodplain core samples will be collected along each  
24 representative reach. The number of floodplain core samples is dependent upon the number of in-  
25 channel samples collected (maximum of 25 in-channel plus floodplain samples per study reach),  
26 the length of the study reach, and complexity of floodplain features. Locations of floodplain core  
27 samples will be determined in manner similar to that done for the USEPA/USGS Cherokee  
28 County, Kansas floodplain study. A transect will be established generally perpendicular to the  
29 stream. The width of the flood plain along the transect will be estimated from topographic maps  
30 and NRCS soils maps, and will include soils that are defined by NRCS as “frequently flooded”,  
31 where the probability of flooding in any year is greater than 50 percent, or “occasionally flooded”  
32 where the probability of flooding in any year is 5 to 50 percent (Natural Resources Conservation  
33 Service, 2002) and verified in the field. Core samples will be collected at equal-spaced intervals



1 across the floodplain and samples designated with a “FP” prefix. Spacing between cores will be  
2 established by dividing the floodplain width (W) by (n+1) where n is the number of core samples  
3 to be collected (generally 5). Core samples will be numbered sequentially from the right edge of  
4 the floodplain (looking downstream) and the first core sample site will be approximately  $W/(n+1)$   
5 ft from the right edge of the flood plain. Spacing may be adjusted depending upon access,  
6 landowner permission, and other on-site considerations. If the floodplain appears complex with  
7 obvious old channel meanders, it is possible that two transects will be done across the floodplain  
8 with 3 to 5 cores per transect. Several bias floodplain cores also may be taken in unusual features  
9 such as old channel meanders or other possible depositional areas. Bias floodplain core samples  
10 will be designated with a “FB” prefix and numbered in the order collected. The anticipated depth  
11 of floodplain cores is to refusal but not to exceed 16 ft. The 16 ft baseline is based on the  
12 maximum depth of core from the USEPA/USGS Cherokee County, Kansas Spring River  
13 floodplain study (Kyle Juracek , U.S. Geological Survey, oral communication, 2010).

#### 14 15 2.2.3 Task 3 Determination of streambed sediment depth

16 Estimates of the depth of unconsolidated sediments in the stream channel will be made along  
17 each representative reach studied. For transect sampling, a tile probe will be used to probe to  
18 refusal at 5 to 10 locations along each transect, including at each transect sample site, and at  
19 longitudinal sample sites, to provide perspective on the adequacy of either the sampling method  
20 or the tile probe to characterize the depth of the sediments. In the case of bias sampling of in-  
21 channel sediments, a tile probe will be used at several locations at each bar where samples are  
22 collected, including at each sample site. Tile probes have been recently been used in Missouri and  
23 Oklahoma to estimate the depths of unconsolidated sediments in streams and tailings near mine-  
24 waste dumps (Pavlowsky and others, 2008; CH2MHill, 2009). A unique ID number will be  
25 assigned to each tile probe location and the horizontal and vertical coordinates will be obtained  
26 with DGPS. Tile probe data will be used to make a rough estimate of in-channel sediment  
27 thickness and volume of bars within the study reach. To supplement transect and longitudinal tile  
28 probe data, the average height of bars that were identified and digitized during the initial GIS  
29 mapping will be estimated using a DGPS.

#### 30 31 2.2.4 Field GPS survey

32 A centimeter-accurate DGPS will be used to provide coordinates and elevations for each in-  
33 channel and floodplain sample and tile probe site. Several bars or riffles along each study reach

1 will be mapped in greater detail (point spacing about 25 ft) using the DGPS to obtain horizontal  
2 and vertical coordinates. The average height of these same bars above the water surface will be  
3 estimated for later use in estimating volumes of sediment, and for comparing the accuracy of the  
4 size of these features to values obtained from digitizing of the NAIP imagery.

## 6 **2.3 Sample Handling and Field Processing**

### 8 2.3.1 Sample collection

9 For both in-channel and floodplain sampling, the primary method for collection of core samples  
10 will be direct-push using truck, tractor, or ATV mounted Geoprobe™ type drilling system. A  
11 wide variety of sampling devices, core tube sizes, slit spoons, Shelby tubes, etc., are available for  
12 use with direct-push drilling. Saturated alluvial sediments, especially gravels and sands, are  
13 typically the most difficult sediments from which to collect continuous core samples. There are  
14 two primary concerns when coring in saturated sediments. One concern is retention of the core  
15 inside the sampling tube or device. Typically this is done using a plastic or stainless steel “sand  
16 catcher” placed behind the core bit (consisting of a circular array of curved plastic or steel  
17 “fingers” pointed upward) to hold the core inside the sample tube when the tube is removed from  
18 the subsurface. The second concern is feeding of sediments into the sampling tube. Oversized  
19 cobbles or debris larger than the opening of the sample tube will block the end of the tube  
20 resulting in refusal or prevent smaller sediments from feeding into the sample tube. This is a more  
21 common problem and source of poor core recovery. Gravel bars typically are armored at the  
22 surface with larger cobbles or contain cobble beds at depth. If core recovery is poor, larger  
23 sample tubes will be used or auger methods will be used to advance through cobble beds. When  
24 core tubes are removed from the saturated sediments, the borehole will collapse preventing  
25 collection of core from deeper depths. One solution is to use a “discrete” core tube that has a plug  
26 at the bottom that can be driven to the desired depth, after which the plug is removed, and the  
27 tube driven further

28  
29 Core diameter will be determined by the largest diameter of the sediments. Continuous core tubes  
30 suitable for discrete sampling are less than 3.0-in. diameter. If larger sized sediments are  
31 encountered then larger diameter core tubes such as split spoon or “Shelby tube” samplers may be  
32 used. A disadvantage of larger core tubes is that disposable liners may not be available (requiring  
33 considerable decontamination between samples) and they may not be suitable in remote areas

1 where hand operated equipment is used or where coring must be done from a boat. In addition,  
2 split spoons are not discrete devices and must be advanced inside augers or an open ended casing  
3 where soft or saturated sediments would cause borehole wall to collapse. Rather than a  
4 continuous core, these methods result in a series of sampled intervals that may have gaps where  
5 samples are not available.

### 7 2.3.2 Other sediment sample collection methods

8 Many sample locations will not be accessible by truck, tractor, or ATV mounted drilling  
9 platforms, and hand-operated methods will be required. Hand-operated methods will employ a  
10 variety of hammer/vibratory/auger, and shovel methods; however, the available options for core  
11 sample collection and penetration depth are reduced. In these areas, the maximum target depth  
12 anticipated is about 5 ft deep.

#### 14 2.3.2.1 Vibro-core methods

15 Hand methods will use a 35 lb (pound) hammer drill, a 5-hp gasoline powered concrete shake, or  
16 small air or hydraulic hammer to vibrate core tubes or other devices into the in-channel  
17 sediments. This method has been used by the USGS to collect shallow sediment samples from  
18 Grand Lake-O-Cherokees in Oklahoma and by the USGS to collect sandy streambed sediment  
19 samples from streams in the Coeur D' Alene mining district in Idaho (Box and others, 2001).

20  
21 Vibro-core drilling is particularly well suited to core sampling in sandy unconsolidated sediments  
22 that are water-saturated. A vibrating drill pipe agitates the intergranular pore water, which lifts  
23 and separates sand grains, allowing downward penetration of the drill pipe. A gasoline powered  
24 concrete vibrator or hammer drill is clamped to the pipe. Eccentric rotation of the vibrator creates  
25 very strong vibrations, which are transmitted to the drill pipe. This method works well but as the  
26 pipe fills its vibration is dampened causing decreasing drill rates and refusal. Before the tube is  
27 withdrawn, a rubber plug is placed in the top to create a near vacuum as the tube is removed.  
28 Sandy sediments tend to fall out of the tube bottom resulting in loss near the bottom of the core  
29 run. Once extracted, the tubes will be transported to a work area on the bank. Samples can be  
30 removed from the tube by either tipping the tube at an angle and lightly tapping to allow the  
31 material to slump out onto a plastic sheet or tray (wallpaper tray) or the tube can be cut  
32 lengthwise with a battery-powered saw.

33

1 2.3.2.2 Auger-Vibro core

2 Modifications to the vibro-core methods likely will be necessary. If refusal is encountered  
3 because the tube becomes filled with sediment then the sediment inside the tube can be sampled  
4 and removed with hand bucket augers and piston samplers, then the tube advanced further into  
5 the bottom. Typically, the overlying water must be removed with a peristaltic pump or bilge  
6 pump. A section of sediment in the tube is removed with a bucket auger, then a hand driven  
7 piston sampler can be used to collect a samples from discrete intervals inside the tube and the  
8 process repeated. The upper one-third to one-half of material in the bucket auger or piston  
9 sampler would be discarded as waste and only the bottom part retained as the field sample.

10  
11 2.3.2.3 Auger-hand core

12 Traditional hand cores (bucket auger with piston core samplers) cannot be used where saturated  
13 sediments collapse the borehole. However, these methods can be used inside casing. At some  
14 locations, a hollow stem auger (HSA) may be turned into the sediments to a specified depth. The  
15 HSA would have a center plug that would be removed allowing a bucket auger or piston sampler  
16 to be turned or driven in front of the auger to obtain a discrete sample. The HSA could be  
17 advanced several feet further, the internal material removed with a bucket auger to the bottom of  
18 the HSA, then a piston samples advanced again. The upper one-third to one-half of the piston  
19 samples would be discarded to waste and only the bottom section retained as actual samples. The  
20 HSA would be advanced using a gasoline-powered hydraulic two-person boring device.  
21 Additional sampling devices, such as the discrete core tube used with the drill rigs can be  
22 advanced up to 4-foot through the HSA using either the vibrator or hammer drill. This is the  
23 preferred method as the core tube has a liner minimizing the potential for cross-contamination.

24  
25 2.3.2.4 Soft sediment piston cores or dredges

26 In areas of greater than about 6 ft in smaller streams that are accessible only by small john boat,  
27 the vibro-core unit cannot used, and a traditional drop-type piston core will be used to collect  
28 samples from the streambed. Penetration depths generally are limited to less than 4 ft, and likely  
29 will be less than 2 ft. These samplers collect a single sample from the bottom and depth of  
30 penetration is dependent upon the substrate bottom. These samplers generally do not have  
31 disposable liners and must be thoroughly decontaminated between samples. If these methods fail,  
32 then the final alternative at these locations is a Ponar or Eckman dredge to collect a limited depth  
33 (less than 0.5 ft) grab sample from the streambed.

1 2.3.2.5 Freeze core

2 If all attempts result in poor or no recovery, two less desirable methods involve freezing the  
3 sediments either inside the sample tube bit (Murphy and Herkelrath, 1996) or onto the exterior of  
4 a steel rod (Knaus and Calhoon 1990). In some cases, core recovery can be enhanced by using  
5 liquid carbon dioxide or nitrogen to freeze the core tube bit. This method is limited to within a  
6 few feet of the water table or water surface. Traditional core tubes can be advanced as described  
7 in above sections, but a special bit and steel tubing is added to the outside of the core tube. Once  
8 the core tube is advanced to depth, liquid carbon dioxide is pumped down the tubing to freeze  
9 sediment inside the core bit. This method is applicable where the sediment can be penetrated but  
10 slumps out of the core tube when the tube is removed from the sediment.

11  
12 A second method is to freeze the fine-grained material to the exterior of a tube that is then  
13 removed from the subsurface. In this method a small diameter steel rod (1-in. diameter or less) is  
14 driven into the sediment, a copper tube is inserted inside the rod, liquid carbon-dioxide or  
15 nitrogen is bubbled inside the rod to freeze sediments to the outside of the rod, and the rod is  
16 pulled. This method results in some recovery of fines (clay up to small sand-sized particles) but  
17 does not produce high quality samples from discrete depths

18  
19 2.3.2.6 Trenching

20 If previous attempts result in little or no core recovery, sediment samples from bar or shallow  
21 riffles accessible by a tractor will be collected from a trench using a backhoe or trencher. Because  
22 of the scale of this method, only a few locations will be sampled and those will probably be  
23 restricted to accessible gravel bars or areas above the water surface. The approach will be used to  
24 excavate a trench in 1 to 2 ft depth increments. At the desired depth, material from one backhoe  
25 bucket or trencher will be dumped onto a plywood or metal sheet. Large cobbles will be removed  
26 by hand, and a shovel used to mix the remaining material. Ten to 15 subsamples will be collected  
27 using a plastic or stainless steel scoop passed through the entire depth of the pile at each location  
28 and composited into a clean plastic tub.

29  
30 2.3.2.7 Hand Shovel

31 Because of access difficulties or problems getting good recovery using the above methods, it may  
32 be necessary to use a hand shovel to sample the sediment. In a similar manner as using a backhoe,  
33 the bar would be excavated by hand in 1 to 2 ft depth increments, the sediment placed on  
34 plywood or a metal sheet, and a composite sample collected from 10-15 subsamples.

1

## 2 **2.4 Borehole Geophysics**

3 To examine the potential for indirect methods of characterizing mine wastes in the subsurface, a  
4 downhole electrical conductivity (EC) and resistivity probe will be evaluated. The probe will be  
5 advanced with the ATV, tractor, or truck mounted direct-push drill rig. As the probe is pushed  
6 into the subsurface, a current is sent through the sediments between two probe contacts. This  
7 current is measured along with the resulting voltage. The conductivity is a ratio of current to  
8 voltage times a constant. The conductivity is different for each type of media with finer grained  
9 sediments, such as silts or clays, having higher electrical conductance values. While coarser  
10 grained sediments, sands and gravel, will have lower values. The EC tool can be calibrated to  
11 soils in an area by matching the EC values with grain-size data of sediments obtained from core  
12 samples. While the usefulness of the EC probe in mapping general characteristics of soils (grain-  
13 size of sediments) is documented, it is not known whether sediments containing mine tailings  
14 may have a unique or anomalous EC signature possibly resulting from sulfide minerals or higher  
15 conductance interstitial water associated with oxidation of sulfide minerals. Provided core sites  
16 encounter obvious (in both appearance and XRF screening data) mine tailings that are in an  
17 accessible area, the EC probe will be run through the mine tailings sediments and nearby non-mine  
18 tailings sediments and the results compared.

19

## 20 **2.5 Sample Handling and Processing**

21

22 Procedures used for the handling and processing of sediment samples will be similar to those  
23 used for the USEPA Cherokee County Floodplain study to allow combining of the two data sets.  
24 Sediment samples will be collected using one of the methods and equipment discussed in section  
25 2.3. Where possible, cores will be collected in 4-ft increments using disposable plastic core tubes  
26 to hold the core inside the sample tube or split spoon, as was done in the USEPA Cherokee  
27 County Floodplain study.

28

29 Because the core barrels are lined with plastic liners, potential contamination of the core  
30 generally is restricted to the core bit and that resulting from handling and storage of the core once  
31 the tubes are opened. Between each core run, the core barrel and bit will be washed with a  
32 laboratory detergent solution and a brush, rinsed in tap water, and sprayed with a deionized low  
33 pressure washer.

1  
2 Grab samples using dredges, shovels, trenching, freeze-core scrapping, etc., will be homogenized  
3 prior to field XRF screening or sampling for laboratory analyses. Homogenization will include  
4 removing large cobble-size rocks or debris, and placing the samples in a plastic or stainless flat  
5 bottom container or flat plastic sheet or cheese-cloth to dry. After drying, the material will be  
6 mixed and a 5-10 gram (g) subsample removed, re-mixed, and the process repeated until a 50-100  
7 g composite sample is collected. The composite sample will be placed in a plastic bag,  
8 disaggregated, and placed flat on a table for screening using the XRF. Five individual XRF scans  
9 will be made on the sample with one in the center and four in each quadrant of the sample. Each  
10 individual result will be recorded. One laboratory confirmation per sample will be collected by  
11 drawing 1-2g subsamples using a plastic or stainless steel spatula (bag mixed between each  
12 subsample) to obtain a 25 g sample. The samples will then be shipped overnight to the USGS  
13 contract soil geochemistry laboratory for geochemical analyses.

14  
15 A field log of each core or sample (depth, core recovery, drilling conditions, date, time, method,  
16 equipment used etc.) will be made at the time of drilling. Each core tube will be capped, labeled,  
17 and shipped to an offsite storage and processing location (Missouri or Oklahoma USGS office or  
18 U.S. FWS office in Columbia, Missouri). Processing will include making a geologic log, air  
19 drying, photographing, XRF screening, and sub-sampling of selected intervals for laboratory  
20 confirmation analysis. For each core, a geologic log describing depth intervals, core recovery,  
21 description of texture, grain size, color, moisture, etc. will be completed and the core sections  
22 photographed as part of the record for each site. As part of the core processing, the core will be  
23 subdivided into 1 ft depth intervals for XRF screening by placing small plastic tabs every foot.  
24 Non-core samples will also be described in a similar manner as core samples.

25  
26 One laboratory confirmation sample will also be collected from each core after XRF screening.  
27 The laboratory sample will be collected using a plastic or stainless steel spoon. The spoon will be  
28 cleaned before each reuse. Less than 25 g of sediment are required and the length of the sample  
29 interval will be varied such that no more than one-half of the core is removed. It is anticipated  
30 that the sample interval will be about 0.5 to 1.0 ft long. The sampled interval will be selected to  
31 avoid sampling across obvious lithologic breaks and avoid large cobbles. The sample for each  
32 core interval will be dried, sieved through a No. 1 sieve, and homogenized as described in section  
33 2.6.1.

1 Chain-of-custody (COC) protocol will be established and followed, as described in section 2.8.

2

3

## 4 **2.6 Analytical Methods**

5

### 6 2.6.1 Metal screening by X-Ray Fluorescence (XRF)

7 After geologic logging, the core will be air dried and analyzed by direct XRF for target mining-  
8 related metals (Pb, Zn, and Cd) and calcium (Ca). The USFWS will supply the XRF and trained  
9 operator. XRF screening will be done at the USGS office in Rolla, Missouri or Oklahoma City,  
10 Oklahoma. The XRF screening will be done at intervals of 3 measurements per 1 ft section of  
11 core. Each 1-ft deep interval (previously marked by the geologist) will be sub-divided into 3  
12 equal length sections and the XRF scan done at the middle of each 1/3 foot interval (fig. 4). This  
13 protocol was used successfully in the screening of floodplain core samples collected from the  
14 USEPA Cherokee County, Kansas project.

15

16 The primary concern for in-situ screening of the in-tact core is errors introduced because of  
17 heterogeneity of the core (both grain size effects and unequal distribution of metals within the  
18 core) and differences in moisture content. To control error from moisture differences, cores will  
19 be air dried until the surface is dry to the touch which may require several days before screening).  
20 If on-site screening of the damp or wet core is done at the field site to guide field sampling  
21 efforts, a re-screening of dried core will be done and the re-screening values will be considered  
22 the final values. Little can be done to control error introduced by heterogeneity of the core, except  
23 to avoid scanning large cobbles and unsure a regular surface to the extent possible. A decision  
24 may be made at the time of screening the core to collect subsamples for processing and  
25 subsequent XRF analysis in the Rolla or Oklahoma City USGS laboratory as described below for  
26 non-core samples.

27

28 **Figure 4.** Generalized X-Ray fluorescence (XRF) screening approach for a sediment core

29

30 As a quality check on the precision of the XRF instrument, during screening on in-tact core, three  
31 repetitive scans at a fixed location will be done at a rate of about once every 15 to 20  
32 measurement intervals (each repeat measurement recorded) and the average reported for that



1 interval. This some procedure will be done for samples that were collect my method other than  
2 intact core (see following paragraph). As a quality check on the variability of the XRF  
3 measurements from the in-tact core, after the triplicate scans are done, three additional scans will  
4 be done at the same interval but the instrument rotated about 120 degrees between each scan.  
5 These three results also will be individually recorded and their average and range compared to the  
6 average and range of the triplicate scans. Instrument scan time is proposed to be 60 to 90 seconds.

7  
8 For samples that cannot be obtained from intact core, bulk samples collected in plastic ziplock  
9 bags will be transported to the USGS laboratory in Rolla, Missouri or Oklahoma City, Oklahoma  
10 where they will be processed and analyzed by XRF. Processing will include verification that  
11 sample bag labeling is consistent with field notes. Samples will be air dried overnight in an oven  
12 at 60 °C. After drying, large obvious cobbles, sticks, pieces of debris such as asphalt will be  
13 removed then the sample disaggregated with a mortar and pestle (if needed), and sieved through a  
14 No. 1 (25.4 mm open size) stainless steel sieve to remove large cobbles and rock fragments  
15 atypical of mining wastes. The sieved material will be collected in a plastic bowl and 100-200 g  
16 removed and homogenized according to Method 6200. A subsample from the homogenized  
17 material will be analyzed using the portable XRF (three scans per sample with individual values  
18 recorded and averaged). Any laboratory samples for ICP-AES analyses will be collected from the  
19 homogenized sample and the remaining homogenized material archived in a clean plastic bag. A  
20 second set of XRF measurements (three scans per sample) will be made on the exact sample to be  
21 shipped for fixed laboratory analyses by ICP-AES.

22  
23 The XRF will be operated by USFWS in accordance with standard procedures described in  
24 USEPA Method 6200. Before analyses of environmental samples, a daily energy calibration  
25 check, and operational checks will be done in as described in Method 6200 and the manufacture  
26 operating instructions. Instrument blanks, calibration verification checks (using ASTM standard  
27 reference samples), and replicate sample, will be run every 10 to 20 samples. The replicate  
28 sample will be analyzed a minimum of 7 times to establish the precision of the XRF.

### 29 30 2.6.2 Laboratory Analyses

31  
32 One laboratory confirmation sample will be collected from each core (maximum of 25 samples  
33 per stream reach studied). Laboratory confirmatory samples will be selected to represent a range

1 of metal (primarily Pb) concentrations determined by XRF screening. These samples will be  
 2 dried, sieved (No. 1 slot), and homogenized as described in section 2.6.1 and discussed in Method  
 3 6200 and placed in clean sealed plastic bags. Before shipping, each sample will be placed flat on  
 4 a table for an additional XRF screening. Five individual XRF scans will be made on the sample  
 5 with one in the center and four in each quadrant of the sample. Each individual result will be  
 6 recorded and the values averaged. After AXF screening, the samples will be submitted to the  
 7 USGS Central Region Mineral Resources Geochemistry (CRMR) laboratory in Denver, Colorado  
 8 for processing. The CRMR laboratory will login the sampling into a Laboratory Information  
 9 Management System (LIMS), and process the samples for subsequent elemental analysis.  
 10 Processing will include drying, disaggregating, splitting, archiving, and grain-size determination  
 11 (sand, silt, and clay-size fractions). Upon receipt the bulk samples will be dried then split with  
 12 one half being archived. The remaining split will be split again and one split sieved to obtain a  
 13 fine (less than 63-micron) fraction and the remaining split weighed, and sieved to determine the  
 14 percent by weight of sand, silt, and clay-size fractions then recombined into a single bulk fraction.  
 15 Each of these splits (fine and bulk) will be submitted to a USGS contract laboratory for routine  
 16 analysis for total (4-acid) digestion and elemental analysis by Inductively-Coupled Plasma  
 17 Atomic Emission Spectroscopy (ICP-AES). The ICP-AES elements analyzed and reporting limits  
 18 are listed in table 2.

20 **Table 2.** List of target elements to be determined by Inductively-Coupled Plasma Atomic Emission  
 21 Spectroscopy (ICP-AES) in laboratory samples.

| Element, and abbreviation | Units          | Concentration Range |        |
|---------------------------|----------------|---------------------|--------|
| Aluminum, Al              | Weight percent | 0.005               | 0.5    |
| Calcium, Ca               | Weight percent | .005                | .5     |
| Iron, Fe                  | Weight percent | .02                 | .25    |
| Potassium, K              | Weight percent | .01                 | .5     |
| Magnesium, Mg             | Weight percent | .005                | .05    |
| Sodium, Na                | Weight percent | .005                | .5     |
| Phosphorous, P            | Weight percent | .005                | .5     |
| Titanium, Ti              | Weight percent | .005                | .25    |
| Arsenic, As               | mg/kg          | 10                  | 50,000 |
| Barium, Ba                | mg/kg          | 1                   | 35,000 |
| Beryllium, Be             | mg/kg          | 1                   | 5,000  |
| Bismuth, Bi               | mg/kg          | 50                  | 50,000 |

| Element, and abbreviation | Units | Concentration Range |         |
|---------------------------|-------|---------------------|---------|
| Cadmium, Cd               | mg/kg | 2                   | 25,000  |
| Cerium, Ce                | mg/kg | 5                   | 50,000  |
| Chromium, Cr              | mg/kg | 2                   | 25,000  |
| Cobalt, Co                | mg/kg | 2                   | 25,000  |
| Copper, Cu                | mg/kg | 2                   | 15,000  |
| Europium, Eu              | mg/kg | 2                   | 5,000   |
| Gallium, Ga               | mg/kg | 4                   | 50,000  |
| Gold, Au                  | mg/kg | 8                   | 50,000  |
| Holmium, Ho               | mg/kg | 4                   | 5,000   |
| Lanthanum, La             | mg/kg | 2                   | 50,000  |
| Lead, Pb                  | mg/kg | 4                   | 50,000  |
| Lithium, Li               | mg/kg | 2                   | 50,000  |
| Manganese, Mn             | mg/kg | 4                   | 50,000  |
| Molybdenum, Mo            | mg/kg | 2                   | 50,000  |
| Neodymium, Nd             | mg/kg | 9                   | 50,000  |
| Nickel, Ni                | mg/kg | 3                   | 50,000  |
| Niobium, Nb               | mg/kg | 4                   | 50,000  |
| Scandium, Sc              | mg/kg | 2                   | 50,000  |
| Silver, Ag                | mg/kg | 2                   | 10,000  |
| Strontium, Sr             | mg/kg | 2                   | 15,000  |
| Tantalum, Ta              | mg/kg | 40                  | 50,000  |
| Thorium, Th               | mg/kg | 6                   | 50,000  |
| Tin, Sn                   | mg/kg | 50                  | 50,000  |
| Uranium, U                | mg/kg | 100                 | 100,000 |
| Vanadium, V               | mg/kg | 2                   | 30,000  |
| Ytterbium, Yb             | mg/kg | 1                   | 5,000   |
| Yttrium, Y                | mg/kg | 2                   | 25,000  |
| Zinc, Zn                  | mg/kg | 2                   | 15,000  |

1

2 All sediment samples not consumed during analysis (core, grab, or otherwise) will be archived for  
3 the duration of the project at either the USGS office in Oklahoma City or Rolla. The final  
4 disposition of the samples beyond the project duration has not been determined.

5

## 6 **2.7 Quality Issues**

### 7 2.7.1 Quality Control

8 Quality control of sampling site variability and analytical determinations of trace metals in soil  
9 will be maintained through the use of replicate samples and standard reference samples. Within

1 site variability in trace metal concentrations will be evaluated through the collection of  
2 sequential-replicate samples or measurements. Relative percent differences will be calculated for  
3 each replicate pair and evaluated against acceptance criteria discussed below. Split-replicate  
4 samples will be used to evaluate the precision of analytical procedures. Standard reference  
5 samples will be used to evaluate the accuracy and bias of analytical procedures. The number of  
6 quality control samples submitted for analysis will equal about 10 percent of the number of  
7 environmental samples.

8

### 9 2.7.2 Data Quality Objectives for Measurement Data

10 Valid data of known and documented quality are needed to meet the objectives of the project. The  
11 majority of concentrations of selected trace metals sediment samples collected for this study will  
12 be determined by portable XRF screening and compared to concentrations in sediment from non-  
13 mined areas to assess the depth and extent of metal contamination from mining activities along  
14 the study reaches. Background concentrations of metals will be values reported in Pope (2005)  
15 and Juracek (2006). In addition, concentrations of selected metals in samples collected and  
16 analyzed by laboratory methods used during this study may be compared to sediment-quality  
17 guidelines to determine potential environmental effects of metals in these sediments. Data quality  
18 indicators include precision, accuracy, representativeness, comparability, and completeness.  
19 Measures of these indicators will be used to validate the data collected for this project.

20

21 **Precision.** Precision is the measure of agreement among replicate measurements of the same  
22 property. In this project, precision of the XRF screening data will be evaluated by making a  
23 minimum of seven replicate XRF measurements made at a single point on the core or sample  
24 without moving the instrument as described in Method 6200. One precision measurement will be  
25 made each day of XRF operation. Split-replicate samples will be analyzed for 10 percent of the  
26 field XRF screening samples and laboratory samples submitted. Replicate field XRF screening  
27 will be done by making three repetitive scans as described in section 2.6.1. Precision of  
28 laboratory data will be evaluated by the preparation of blind split samples routinely prepared by  
29 the CRMR Geochemistry laboratory and submitted blindly to the contract analytical laboratory.  
30 Acceptable variability among analyses of split-replicate samples for this project will be a relative  
31 percent difference  $[(A-B)/(A+B/2)]*100$  between replicate pairs (A and B) of plus or minus 20  
32 percent.

33

1 **Accuracy.** Accuracy is the measure of an individual measurement or the average of a number of  
2 measurements to the true value of that being measured. Accuracy includes the combination of  
3 random error (precision) and systematic error (bias) components that may result from the  
4 sampling and analytical operations. Accuracy of the XRF screening will be measured by analysis  
5 of standard reference samples. Confirmation of XRF accuracy and overall XRF analyses of  
6 sample handling methods will be evaluated by analysis of split samples between the XRF and the  
7 analytical laboratory, where the laboratory value is assumed to be the “true” value. Before a  
8 laboratory sample is shipped, 5 individual XRF scans will be done as described in section 2.6.2.

9  
10 Accuracy of laboratory analyses will be determined by the analysis of standard reference samples  
11 of sediment with known concentrations of selected trace metals. Acceptable variability among  
12 analyses of standard reference samples will be within the published limits for each constituent for  
13 each standard or plus or minus 20 percent whichever is greater, except when constituent  
14 concentrations are at or near analytical detection limits.

15  
16 **Representativeness.** Representativeness is the degree to which data accurately and precisely  
17 represents a characteristic of a population parameter at a sampling location. The  
18 Representativeness of the XRF data will be evaluated by rotating the instrument about a fixed  
19 point and making three replicate measurements at the same depth interval as described in section  
20 2.6.1. Comparison of the average, range, and standard deviation of these three measurements will  
21 provide an assessment of the ability of sample collection to adequately describe the average  
22 concentrations of selected trace metals in the sample. Because of variable nature of metals in  
23 sediment (typically associated with small metal-rich mineral grains such as galena) the acceptable  
24 variability among analyses of sequential-replicate samples will be a percent relative standard  
25 deviation (RSD) of plus or minus 40 percent.

26  
27 **Completeness.** Completeness is the measure of the amount of valid data obtained from a  
28 measurement system as expressed as a percentage of the number of valid measurements that  
29 should have been collected. This includes the collection, proper handling and labeling, shipping,  
30 and analysis of the samples. To generate data of the quantity necessary to meet the objectives  
31 stated for this project, 70 percent of the data in the designed assessment system should be  
32 collected and analyzed. This unusually low percentage is because of the high density of data  
33 proposed to be collected and because of the unknown nature of the sediments and difficulty in  
34 obtaining sediment samples from a river environment. In addition, the desired completeness ratio

1 may be modified because of the variable physical or environmental characteristics at the time of  
2 sample collection. Any deviation from a 70 percent completeness ratio will be evaluated in  
3 context of all other available data before the decision is made that missing data have irreparably  
4 affected the potential for the project to meet the stated objectives.

5  
6 **Comparability.** Comparability is a measure of the confidence with which one data set or method  
7 can be compared to another. For this project, comparability will be addressed through the use of  
8 common and accepted practices in sample collection and analysis and by reporting data in  
9 standard units. The preparation, splitting, and analysis of laboratory samples for this project will  
10 be done as part of the routine USGS geochemistry surveys done by the CMRA team and  
11 compatible with thousands of samples analyzed from across North America. In addition, the  
12 comparison (regression analysis) between the XRF screening of laboratory samples prior to  
13 submittal with the bulk laboratory data will enable comparison between the XRF and total  
14 digestion and ICP-AES analytical data.

## 15 16 **2.8 Documentation**

17 The records for this project will include miscellaneous correspondence, field logs or notebooks  
18 and field data work sheets, laboratory analytical reports, field activity documents, quarterly  
19 progress reports, and the final report prepared at the conclusion of this project. Field bound  
20 notebooks will be kept that will document the general daily field activities. Field logs will include  
21 observations about weather and physical conditions of the study reach, descriptions of specific  
22 sampling sites, descriptions of field visits (reconnaissance, utility clearances, sampling activities),  
23 personnel involved, and contacts with landowners or other officials. Geologic logs will be kept  
24 for each core or sampling site describing the equipment used, drilling methods, field personnel,  
25 general lithology, recovery, sample depths, etc. Sampling site folders (for each representative  
26 stream reach investigated) will be maintained by the USGS for the duration of the project. These  
27 folders will contain all of the aforementioned documents and will be kept at the USGS office in  
28 Rolla, Missouri.

29  
30 Reports, geologic logs, and results of field XRF screening and laboratory analyses will be  
31 submitted to the FWS Project Manager upon publication or completion. Any other pertinent  
32 observations or deviations from the general SAP procedures also will be recorded on the field  
33 sheets. Field books and field sheets will be signed and dated by the person making the entries.

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In accordance with USGS policy, all data collected as part of routine data collection by the USGS are stored in the National Water Information System (NWIS) computer database. Sample sites are assigned a unique 15 digit station number that generally is a combination of the latitude and longitude of the site to the nearest second plus a 2-digit suffix that is a sequence number from 01 to 99. The field XRF screening data will not be entered into the NWIS, but will be tied to the NWIS station number in the internal project database.

The NWIS is organized by state where the sample site is located, such that the responsibility for uploading and maintaining backups of data stored electronically in NWIS is the responsibility of the USGS state office. The USGS principle investigator (Doug Mugel) will ensure that all sample sites are entered into the NWIS.

The Principal Investigator also will establish, maintain, and document a COC system for field samples that is commensurate with the intended use of the data. A sample is in custody if it is in actual physical possession or in a secured area that is restricted to authorized personnel. Every exchange of a sample between people or places that involves a transfer of custody will be recorded on appropriate forms that document the release and acceptance of the sample. Each person involved in the release or acceptance of a sample will keep a copy of the transfer paperwork. The Principal Investigator or designee is responsible for ensuring that custody transfers of samples are performed and documented according to the requirements listed below:

- The means for identifying custody should be clearly understood (use of forms, stickers, etc.);
- Instructions for documenting the transfer of samples and the person responsible for this documentation must be clearly defined; and
- A plan must be in place for maintaining records in a specific location for a specific period of time (for example, in the site folder).

Detailed guidance on chain-of-custody procedures is provided by the USGS National Water-Quality Laboratory (NWQL) at URL [http://rstalcoarv.cr.usgs.gov/USGS/ASRs/asr\\_instructions.html](http://rstalcoarv.cr.usgs.gov/USGS/ASRs/asr_instructions.html).

Data from the USGS contract geochemistry laboratory are transmitted electronically to the Principal Investigator and entered into the NWIS. Environmental sample data are entered into the NWIS QWDATA database 01 (DB1); QC data are entered into the NWIS QWDATA database 02

1 (DB02). Data entry is the responsibility of the Principal Investigator or designee. The NWIS  
2 QWDATA database receives daily incremental backup and weekly full backup.

## 4 **2.9 Geospatial analysis**

5 A GIS will be used to organize all data collected in this study. All sample sites will have GPS  
6 coordinates and a unique ID number to allow spatial analysis of the data. In addition to its use for  
7 organizing and simple mapping and overlaying of sample sites on current and historical images,  
8 soils maps, etc., the GIS will be used to estimate sediment thickness stored within the current  
9 (2010) stream channel along each study reach. Two methods for estimating these thicknesses will  
10 be compared. First, streambed and gravel bar areas along the study reach will be estimated by  
11 digitizing from the NAIP imagery and combined with tile probe depths and depths of  
12 contamination to provide thickness estimates of contaminated streambed or bar sediments.  
13 Generally, the elevation of the maximum depth of the tile probe along the reach (adjusted for  
14 channel slope) will be used to establish a base plane that will be assumed to be the base of the  
15 channel sediments. Second, the thickness of selected bars will be estimated using the base plane  
16 as above and the footprint and elevations from the DGPS survey. Direct comparison of these two  
17 methods will determine if methods can be optimized and quickly re-scaled to much larger stream  
18 lengths across the TSMD, or what additional low-density data might be needed along the streams.  
19 This data may also be used to estimate volumes of contaminated sediments within the stream.

## 21 **3.0 Deliverables and Schedule**

### 23 **3.1 Deliverables**

24 The USGS will prepare a progress reports to the FWS project manager each calendar quarter. The  
25 reports will describe general project activities, and scheduling. Reports will be submitted monthly  
26 during periods of intense field activity,

28 A final report USGS Scientific Investigations Report will be produced describing the methods  
29 used, and the spatial and vertical distribution of target metals within sampled reaches of streams  
30 in the Tri-State Mining District. Concentrations of metals will be illustrated in cross sections  
31 showing depth and general lithology. The total volume of contaminated sediment and mass of  
32 target metals (Pb, Zn, and Cd) will be estimated by different GIS methods along each sampled  
33 study reach. Comparison of the in-channel longitudinal and special sample sites to the transect



1 sites, and the use of bias sampling instead of transect sampling will be presented and discussed in  
 2 the context of optimization of a sample design for wider-scale implementation across the TSMD.  
 3 The various sampling methods will be discussed and compared to benefit any future study.

4  
 5 **3.2 Project Schedule**

6 The project funding was secured during fiscal year 2009. The project is anticipated to require  
 7 three years to complete beginning with preparation of the SAP. Initial field reconnaissance and  
 8 selection of representative study reaches is expected to be completed by October 2010, with  
 9 initial field sample collection during December of 2010. Subsequent field work will be  
 10 completed in the spring and summer of 2011. Data compilation and analysis will be conducted  
 11 from winter 2012 through spring 2012. A final interpretive report will be written during 2011 and  
 12 2012, and will be published by March 2013. A summary of the project timeline is shown in table  
 13 3.

14  
 15 **Table 3.** Generalized project timeline.

16

| Task  | Estimated begin date | Estimated completion date |
|---|----------------------|---------------------------|
| Prepare SAP                                       | Feb 2010             | April 2010                |
| Draft SAP review by FWS                           | April-2010           | May-2010                  |
| Revise and prepare final draft SAP                | June-2010            | August-2010               |
| Final draft SAP to FWS                            | Sept-2010            | Sep-2010                  |
| Finalize SAP                                      | Nov-2010             | Nov-2010                  |
| Begin field reconnaissance (phase 1)              | June-2010            | Mar-2011                  |
| Select study reaches and obtain access/clearances | June-2010            | Mar-2011                  |
| Phase 1 field sampling effort                     | Dec-2010             | May-2011                  |
| Laboratory analyses (phase 1 and 2)               | Feb-2010             | Aug-2011                  |
| Phase 2 field sampling                            | July-2011            | Aug-2011                  |
| Data compilation and analyses                     | Jan-2012             | June-2012                 |
| Draft report preparation                          | July- 2012           | Sept-2012                 |
| FWS review of draft report                        | Sep- 2012            | Oct- 2012                 |
| Final Report preparation and publication          | Nov-2012             | Mar- 2013                 |

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