East St. Louis
"Biosolid" Lead Remediation Project

Southwestern Illinois Resource Conservation & Development, Inc.

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Funding Provided By USDA-NRCS

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Executive Summary

The East St. Louis "Biosolid" Lead Remediation Project is an effort designed to demonstrate alternative, cost-effective measures that can be utilized in the remediation of inner-city lead contaminated sites. East St. Louis historically contained a number of paint manufacturers and lead smelting facilities. Today, much of the industry has left the community, and what remains are numerous contaminated sites with a costly clean-up bill.

Southwestern Illinois RC&D, Inc. was asked by US EPA, USDA NRCS, the project funding agency, and the Metro-East Lead Collaborative to implement a demonstration project that would utilize a newer technology in lead remediation, termed bioremediation. In reviewing the potential of this technology we settled on a process that utilizes "biosolids", a combination of composted yard and municipal sewage sludge, along with fertilizers and soil amendments to physically bind the lead to soil particles.

The outcome of this process is not the physical removal of lead from the contaminated site, it is the actual bonding of the lead to the soil particles. In doing so, lead that is ingested by humans is more likely to pass through the digestive system rather than being absorbed into the bloodstream.

Benefits of biosolid lead remediation projects include:

1) Reduced costs associated with lead remediation
2) Reuse of other materials, including yard waste and waste treatment
3) Reduced pressure on landfills
4) The biological nature of the procedure will increase the effectiveness of the process over time.
5) The rate of absorption of lead into the bloodstream is reduced.

This report will highlight the contacts, support documents, equipment, procedures and methods that were utilized in developing our project. A brief discussion regarding the overall costs associated with the project is also included, however it should be recognized that significant variations to the costs could be achieved based on the availability of suitable contractors and or a source of Class "A" biosolid compost.

Historical Overview:

The site chosen for this pilot project is located on the south side of St. Clair Avenue, between 16th and 17th Streets in East St. Louis, Illinois. East St. Louis is a former heavy-industrial community that is now forced to deal with the many sources of contamination that were deposited within the city's soils over the past several decades. As the production of paint, and lead smelting, were historically two prominent industries within this community, one of the main contaminants found in the soil is lead dust/particles.

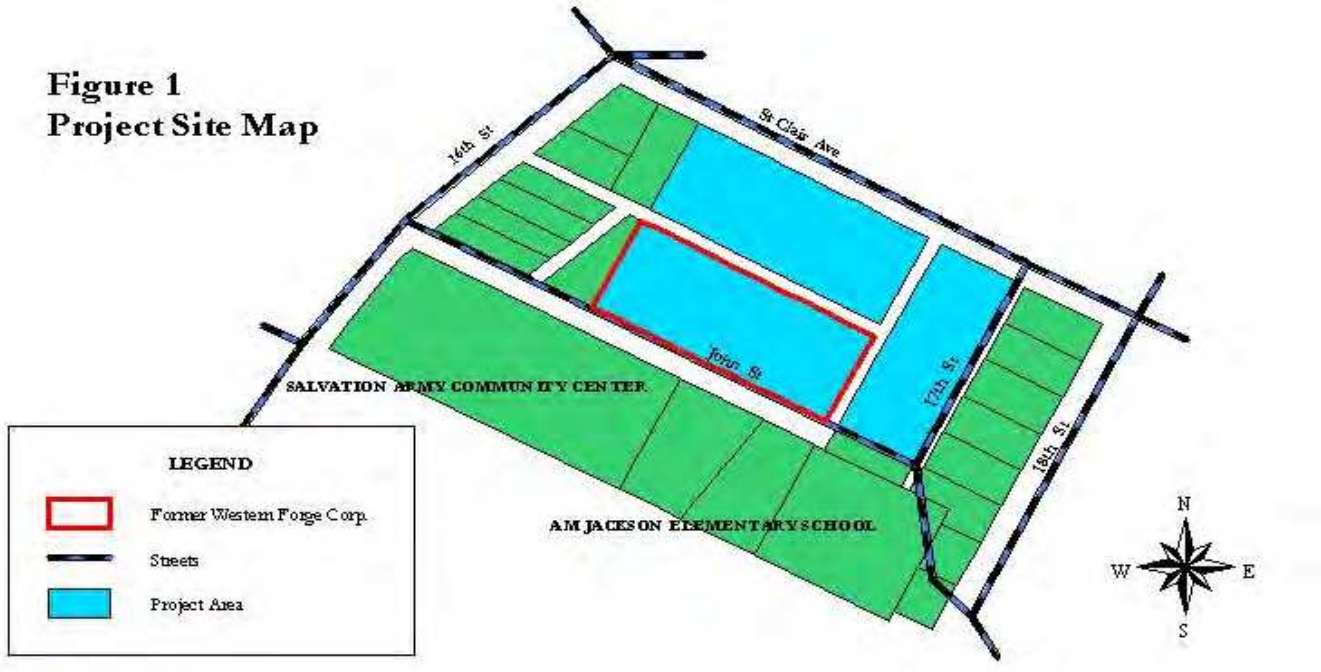
For purposes of this project, the term "site" will identify an area as defined below, see Figure 1.

The site is located in a mixed residential and commercial area. It is immediately visible from St. Clair Avenue, a main community thoroughfare, as well as Interstate 64. The immediate neighborhood surrounding the project site includes A.M. Jackson Elementary School, south and east, an auto repair business, east, a Salvation Army Community Center, south and west, residences, abandoned buildings and vacant lots.

A portion of the project site is the former location of Western Forge Works, (the suspected source of contamination), a metal forging business, and the remainder of the site is the location of former residences. All structures, with the exception of three billboards, have previously been removed from the immediate project area.

A total of five entities control current ownership of the 2-acre (+-) site. Much of this property has been purchased at auction, for back taxes, and there appears to be no immediate plan for the future development of the site, either individually or in collaboration amongst the five entities. Currently, the site receives infrequent mowing, and is therefore mostly devoid of woody material.

Figure 1
Project Site Map



Effects of Contamination:

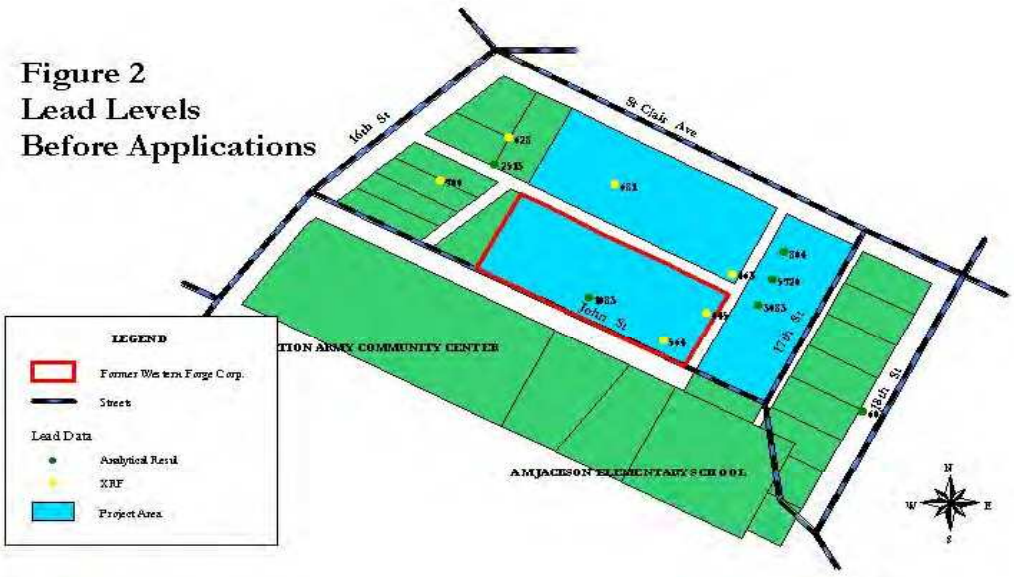
Through random blood screening of children, officials have identified an alarming rate of lead poisoning among the youth within certain neighborhoods within the Metro-East, including many East St. Louis neighborhoods. To determine the cause of this phenomenon, the Illinois Department of Public Health performed random soil testing throughout the city in 1999, and has determined that several sites, predominately bordering former industrial locations, contained lead levels in excess of US EPA guidelines.

The US EPA responded to this information and has prepared a detailed site assessment of several sites within the community, including this project site; Site Assessment Report, Western Forge Works Site, East St. Louis, St. Clair County, Illinois, Tetra Tech EM Inc.

The detailed analysis of this site showed that "XRF screening results showed that at 17 out of 34 screened points (50 percent) at the former metal forging property and at adjacent properties from 16th Street to the west, "old road" to the east, St. Clair Avenue to the north, and "private road" to the south, the 400-mg/kg PRG for lead was exceeded". Further analytical study confirmed this information. See Figure 2.

Prior to the implementation of this project there were no warning signs, fences or other barriers to prevent persons, and particularly youth from the adjacent school and community center, from entering or playing on this site. Furthermore, there were no mechanical means of containing lead to the site and preventing it from leaching into neighboring properties.

In light of this information, the US EPA determined that this site should receive immediate remediation efforts.



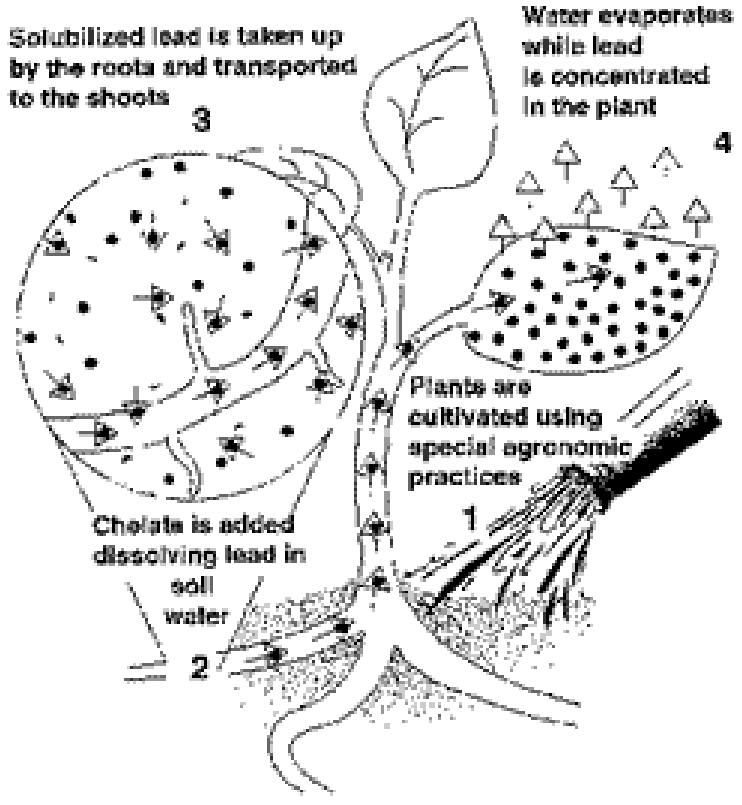
Biosolid Remediation:

In Fall 2000, Southwestern Illinois RC&D, Inc. was asked to participate in the Metro-East Lead Collaborative and implement a pilot project that would utilize alternate practices in reducing human exposure to lead. USDA NRCS agreed to provide funding to this project in the amount of $50,000. Initially it was mutually agreed that the RC&D would concentrate on a process commonly referred to as "phytoremediation".

Phytoremediation is a broad term for a process that utilizes plants to collect contaminants, and the plants then either (1) release broken-down compounds into the environment in a non-toxic form, or (2) the plant is harvested and the plant/contaminant mix is properly disposed of.

While the first process works well with many contaminants, it has not been shown to work with lead. There has been significant work done with plant material that will collect lead and allow for the proper disposal of the contaminant in a landfill, however, there are currently significant limitations in this process, including:

1) Only a limited number, five (5), plants are known to be hyper-accumulators (the ability to collect large quantities, as a percentage of the plant's weight, of the contaminant without succumbing to the toxin) of lead. Each of these plants has an extremely slow growth rate, and would therefore be unacceptable to be used in this process.



2) A chemical referred to as EDTA (chelate) can be utilized in increasing the rate of lead translocation (soil to plant) in other types of plants that produce a large biomass, (corn,

sunflowers and mustard), however lead that the chemical frees up that is not immediately absorbed by the plant is free to leach offsite, or into the immediate groundwater supply.

3) The costs associated with collecting the free lead created by the application of EDTA, (placing contaminated soil into a plastic liner, growing/harvesting the plants, and then replacing the soil) is prohibitive.

A third process within the phytoremediation "umbrella" includes treating lead contaminated sites in-place (in-situ), by creating a physical barrier (turf grass) and also incorporating amendments that promote the formation of insoluble Pb species. In implementing this process, termed "bioremediation", with the right proportion of materials, a tight adhesion can be created that will physically bond the lead to soil particles. In doing so, ingested lead is much more likely to pass through a digestive system, rather that being absorbed into the bloodstream.

As this is a biological process, the physical attraction of the lead to the soil will actually increase over a period of time, making this a very attractive, affordable process to put into practice. The process has been successfully implemented at a number of large-scale smelter sites throughout the country, and we therefore established the following goal to lead this project:

To define the processes and equipment, identifying suppliers and establish evaluation methods that will allow for the implementation of a biosolid lead remediation project on a small-scale, inner-city site in the St. Louis Metro-East area.

Soil testing is an important first step in developing a work plan for this process. Lead levels, pH, organic composition and current nutrient levels will all play a part in the type and rate of material application. In general terms, a mixture of Phosphorous, in a non-acid form, and Calcium Carbonate will be blended into the soil with a Class "A" biosolid. Materials for use as the compost can come from a variety of sources. In our case, a local supply of compost, (40% biosolids, 60% yard waste) was readily available from the City of St. Peters, Missouri.

The rate of application of Phosphorous would equate to obtaining a 2:1 molar ratio of P:Pb in the soil. The Calcium Carbonate application would equate to raising and maintaining the pH of the soil to a minimum of 6.5. Biosolids were applied at a rate of 100 dry tons per acre, or approximately 500 wet tons of material. This should produce a layer of compost approximately 3" deep on the project site.

The incorporation of compost, in conjunction with the Phosphorus and Calcium Carbonate has done an excellent job in reviving a compacted, nutrient-poor site. An excellent stand of turf grass was witnessed within three months of seeding.

Work Plan:

While this process has been completed on a selection of sites throughout the country, our first task included the need to develop a detailed work plan that took into account the size, location and degree of contamination of this particular site. The development of this plan was accomplished with the assistance of the following people:

Mr. Randy Alvey	Alvey Laboratory, Inc.
Ms. Sally Brown, Ph.D.	U. of Washington, College of Forest Resources
Mr. Rufus L. Chaney, Ph.D.	USDA-Agricultural Research Service
Ms. Noemi Emeric	US EPA Gateway Regional Team Manager
Mr. Scott Fredericks	US EPA Environmental Response Team
Mr. Kevin Turner	US EPA Environmental Scientist, OSC

In addition, a number of reports, and or previous case studies, were reviewed for information that could be utilized in the development of our project. A sampling of these reports includes:

1) Li, Y.-M., R.L. Chaney, G. Siebielec and B.A. Kershner. 2000. Response of four turfgrass cultivars to limestone and biosolids compost amendment of a zinc and cadmium contaminated soil at Palmerton, PA. J. Environ. Qual. 29:1440-1447.
2) Chaney, R.L., S.L. Brown, J.S. Angle, T.I. Stuczynski, W.L. Daniels, C.L. Henry, G. Siebielec, Y.-M. Li, M. Malik, J.A. Ryan and H. Compton. 2000. In situ Remediation/Reclamation/Restoration of Metals Contaminated Soils using Tailor-Made Biosolids Mixtures. In Proc Symposium on Mining, Forest and Land Restoration: The Successful Use of Residuals/Biosolids/Organic Matter for Reclamation Activities (Denver, CO, July 17-20, 2000). Rocky Mountain Water Environment Association, Denver, CO.
3) Sterrett, S.B., R.L. Chaney, C.E. Hirsch and H.W. Mielke. 1996. Influence of amendments on yield and heavy metal accumulation of lettuce grown in urban garden soils. Environ. Geochem. Health 18:135-142.
4) Brown, S.L., Q. Xue, R.L. Chaney and J.G. Hallfrisch. 1997. Effect of biosolids processing on the bioavailability of Pb in urban soils. pp. 43-54. In "Biosolids Management Innovative Treatment Technologies and Processes. Proc. Water Environment Research Foundation Workshop #104 (Oct. 4, 1997, Chicago, IL). Water Environ. Res. Found., Alexandria, VA.
5) Solubility/Bioavailability Research Consortium; Standard Operating Procedure: In Vitro Method for Determination of Lead and Arsenic Bioaccessibility. See Attachment I.
6) US EPA, EPA530-F-97-042, October 1997, Innovative Uses of Compost Bioremediation and Pollution Prevention, http://www.epa.gov/epaoswer/non-hw/compost/bioremed.txt
7) US EPA, EPA530-B-98-001, March 1998, An Analysis of Composting as an Environmental Remediation Technology, Chapter 4, pages 1-9, Potential for

Reclamation of Mine Spoils and Brownfields with Compost, http://www.epa.gov/epaoswer/non-hw/compost/analysis.txt

8) Berti, W.R., Cunningham, S.D., Cooper, E.M., 1998; Case Studies in the Field-In-Place Inactivation and Phytorestoration of Pb-Contaminated Sites, Chapter 17.

9) In addition, the following web sites provide additional information regarding biosolid remediation:
http://faculty.washington.edu/clh/bunker.html
http://faculty.washington.edu/clh/leadville.html
http://faculty.washington.edu/clh/wet.html

Pre-implementation Action Items:

Prior to implementing this project a number of action items needed to be addressed to allow for a smooth transition into actual on-the-ground activities. Included were:

1) Southwestern Illinois RC&D, Inc. participated in periodic meetings with the Metro-East Lead Collaborative and the community to inform the group of the status and likely results that would be accomplished through this project.

2) Southwestern Illinois RC&D, Inc. created, produced and disseminated (with the assistance of members of the lead collaborative) approximately 800 copies of an informational flyer that was designed to raise awareness within the immediate neighborhood towards this project. See attachment I. This flyer was distributed at the adjacent school, community center, surrounding neighbors and at a public information meeting.

3) Southwestern Illinois RC&D, Inc. conducted a title search for each property within the project site. Once this task was completed, each landowner was contacted and a signed authorization form was obtained.

4) Southwestern Illinois RC&D, Inc. hired an independent contractor to perform both on-the-ground applications, as well as the pre and post-application testing. A supervisor for this company was required to complete a 40-hour HAZWOPER (Hazardous Waste Operations & Emergency Response) training course. (Equipment needs for the project will be discussed later in this report; testing requirements are outlined in Attachment II.)

5) Southwestern Illinois RC&D, Inc., with the assistance of Scott Fredericks, US EPA, located a source of Class "A" Biosolids (US EPA Guidelines) which contained a mix of yard waste, 60%, and waste treatment facility solid waste, 40%.

6) Southwestern Illinois RC&D, Inc. obtained both a "Building Permit" and "Contractor Certificate of Registration" permits from the City of East St. Louis.

Work Plan Overview and Contractor Commentary:

Alvey Laboratory Inc., Belleville, Illinois was retained by Southwestern Illinois RC&D, Inc. to perform the field phase of this pilot project, researching in-place Stabilization and Biological-deactivation of soil borne lead at the test site. As mentioned earlier, the process is NOT INTENDED to remove either the soil matrix or the actual lead within the soil. The intent is to tightly bind the lead metal to the soil particles consequently reducing biological availability. The reduction in bioavailability will decrease the threat level to local children as well as the environment while utilizing recycled waste materials.

Prior to any on-site activity, multiple sampling locations were determined utilizing D-GPS technology. Pretreatment samples were collected and archived. Follow up samples will be collected over various time lines to determine the degree of success or failure associated with the project. The bioaccessibility value is determined as a factor of an in vitro extract (which indicates the lead fraction which is soluble in the gastrointestinal tract) divided by the total lead. Reducing the bioaccessibility will lessen the threat of lead to people and the environment.

During the initial phase of the on-site cleanup process, multiple areas of dumped garbage were discovered in sub-areas that had not been mowed for multiple years. The initial mowing was achieved by utilizing an 85 horsepower tractor attached to a 9 foot "BUSHHOG" with a stump jumper attachment and heavy-duty uplift blades. At the conclusion of the initial mowing it was determined that the blades were a total loss as the result of damage incurred from hidden concrete and rocks.

Additionally, the first mowing revealed multiple concrete foundations, rock foundations, large loose pieces of dumped concrete and other human generated debris which were previously unknown to exist at the site. Multiple tires, tires with rims as well as salvage lumber were also discovered.

The "junk" lumber, fallen tree branches, discarded utility poles, etc. were placed in a large dumpster provided by Southwestern Illinois RC&D, Inc. The small 64 in3, medium 8 ft3, and large 25 ft3 pieces of concrete and rock were removed and stockpiled in an area to the west which was scheduled for soil removal. Approximately 36 tons of concrete material was removed by hand from the site.

As this was a pilot project it should be noted that this is an unforeseen complication to the project and extremely expensive to correct. In future projects, a contingency allowance must be allotted for unknown and undiscovered debris that must be removed from the site.

The perimeter of the entire site was mowed approximately 25 feet wide in order to provide a work area for the erection of the perimeter fence. The site was encompassed with a four-foot high orange construction fence, with one entrance area gated to allow equipment access to the site.

Initially the area east of the site (area around the billboards) was outside the perimeter fence to facilitate receipt of the compost (18 semi-loads) from St Peters, Missouri. After receipt of 85% of compost, the perimeter fence was moved to encompass the entire area. The gated section was closed at all times when Alvey Laboratory, Inc. staff was not on site.

The construction fence is supported by steel "T" posts placed at approximately ten-foot increments along the perimeter of the fence line. Significant areas of difficulty were encountered on the John Street side of the site due to the presence of asphalt and discarded concrete. After the mowing was completed a temporary gate area was established on the 16th street side of the site to allow for the egress of loads of debris.

A four-foot by six-foot project sign was erected along St. Clair Avenue. This sign identifies the project site and denoted the sponsorship of the project. Additionally, a second sign, two foot x three foot, was placed at the site / elementary school yard interface. This sign brings additional awareness to the site and advises children to refrain from crossing the work area. The RC&D supplied all of the signage for the project.

Alvey Laboratory, Inc. applied 250 pounds per acre of 0-45-0 (triple Super Phosphate) and 4000# per acre of pelletized limestone (Calcium Carbonate) to the test area. Note: The application rates were doubled in the northeast corner of the project site to reflect higher lead levels in this area. After these applications we performed the initial tillage operation.

There was a significant rainfall event shortly before the material applications and tillage so there was no need for a water wagon to control dust at that time. The initial tillage determined that there were several roadways as well as buried foundations that previously had not been identified. We removed as many of the obstacles as possible without utilizing bulldozers or backhoes although we did utilize a 3000# capacity loader tractor for some of the larger pieces.

Following the secondary debris removal (initial tillage with a 9-point chisel plow, debris removal, second tillage with 9-point chisel plow, and secondary debris removal) we spread the stockpiled Class "A" compost as provided by St. Peters, Missouri. The compost was spread utilizing both a "SLINGER" type spreader as well as a conventional end-gate-rotary beater spreader. Application of the compost was at the rate of 400 cubic yards per acre. This generated a "mat" of compost 3-4 inches in depth.

After the initial application of compost St. Peters delivered the remaining balance of the compost. This material was spread on the remaining area of the site. It should be noted that we were unable to accomplish a large amount of tillage in the area immediately around and under the billboards. We found significant concrete slabs and did not want to remove for fear of destabilizing the sign itself.

The majority of the compost "mat" was incorporated into the top 3 inches of soil by disking twice at perpendicular angles. After the disking, complete soil leveling was accomplished with a harrow-type leveling device again operated twice over the site.

We applied 150# per acre of 12-12-12 to the site prior to broadcasting the 425# per acre of Kentucky 31 Fescue as well as 2 bushels of winter wheat. The winter wheat will served to provide a quick green-up to the site. All of these materials were lightly incorporated into the soil again with a harrow-type device.

Several days of precipitation followed the initial seeding. As soon as possible, 15 tons of finely chopped straw was applied to the site. An excellent stand of Fescue as well as winter wheat was evident by late October 2001. All fencing materials were removed in the spring of 2002.

Verification, Bioaccessibility Values:

Objective: To provide analytical information to be used in determining the potential use of procedures outlined in "Pilot Project –Lead Bio-Remediation East St. Louis" by Alvey Laboratory, Inc. The process used to measure this potential is called bioaccessibility. Bioassessibility is a function that compares the *in vitro* lead (that portion that could be absorbed in the gastrointestinal system) verses the total lead in the soil.

Samples: Samples were collected from five (5) different spots on the project site using d-GPS mapping. See Figure III. Initial samples were collected on September 6, 2001. These were pre-treatment samples. Second samples were collected on December 27, 2001. This sample date was approximately two months following treatment.

Procedure: Samples were dried and crushed to pass through a USA Standard 60 mesh testing sieve (250 micrometers). Duplicate samples were analyzed. For total lead digestion, the method EPA 3050A was used. For *in vitro,* the method used was "Standard Operating Procedure: *In Vitro* Method for Determination of Lead and Arsenic Bioaccessibility" by the Solubility/Bioavailability Research Consortium provided by Dr. Sally Brown at the University of Washington.

Test Results:

Sampled before amendment/compost addition:

Site	In vitro Pb (mg/L)	Solid Pb (mg/kg)	Bioaccessibility Value*
1	182.7	189.6	96.4
2	167.8	223.8	75.0
3	292.3	337.3	86.6
4	693.9	1388.3	50.0
5	1221.3	1741.7	70.1
Average	**511.6**	**776.2**	**75.6**

Sampled after amendment/compost addition:

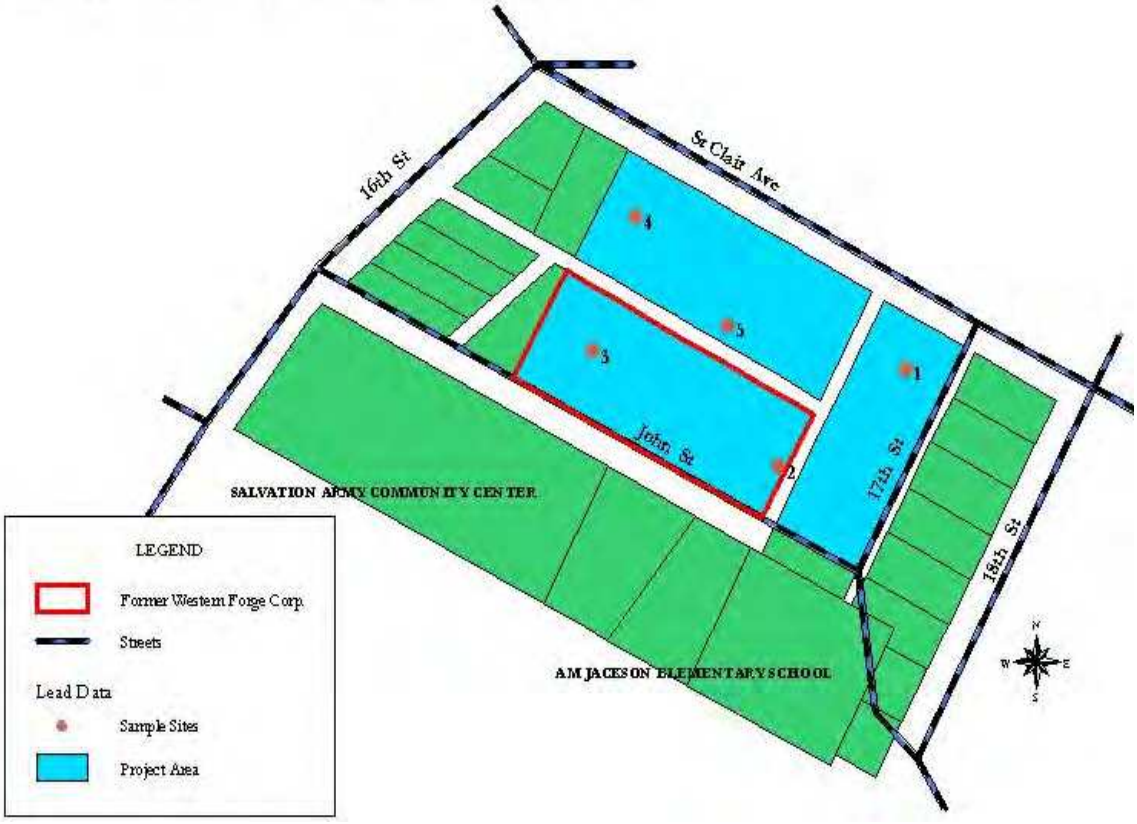
Site	In vitro Pb (mg/L)	Solid Pb (mg/kg)	Bioaccessibility Value*
1	73.8	140.0	52.8
2	96.6	215.1	44.9
3	307.2	288.5	79.1
4	302.3	347.4	87.0
5	436.5	732.5	59.6
Average	**243.3**	**364.7**	**64.7**

* Bioaccessibility value = (in vitro Pb, mg/L)/(solid Pb, mg/kg) x 100

Samples run using SOP from "Solubility/Bioavailability Research Consortium"

Discussion: There is a significant decrease in both the total lead and *in vitro* lead when comparing the values before and after treatment. The bioaccessibility values were also decreased after only two months. Because the compost mixed with the soil procedure is looking at a biological process, additional samples should be taken in the future to follow any potential additional decrease of bioaccessibility values.

Figure 3
E. St. Louis Remediation Project Site
Alvey Laboratory, Inc. Sample Locations



Summary:

The testing and evaluation process utilized for this project is an outcome of the Solubility/Bioavailability Research Consortium; Standard Operating Procedure: In Vitro Method for Determination of Lead and Arsenic Bioaccessibility. (See Attachment II.) Based on a similar case study from Joplin, Missouri, a 30% to 40% reduction in the bioavailability with the in vitro can be expected. Feeding studies on juvenile swine indicate a 38% reduction in swine, which would reflect an approximate 69% reduction in human adults.

While these testing procedures give an accurate representation of the project's current results, it does not reflect changes over time, and as this is a biological process that has been proven to increase in effectiveness over time, we anticipate even greater results in the future.

Utilizing this format, the current testing in conjunction with results obtained in previous animal feeding studies (Joplin, Missouri), suggests that the reductions that we have obtained with the in vitro, combined with the overall dilution of the total lead, indicates that this process may be fully protective of human health.

As the amendments incorporated into the site will work in conjunction with each other over time to increase the benefits of the project, long-term maintenance to the site will be minimal. The following maintenance practices represent those practices recommended for turf grass, and would therefore be beneficial in maintaining a dense barrier between humans and the soil on this site:

1) The site should be mowed on a regular basis (weekly).

2) The site should receive four applications of a balanced turf grass fertilizer on an annual basis.

3) The site should be tested for pH on an annual basis. If the level falls below 6.5, lime (Calcium Carbonate) should be added to raise the rate to neutral (7).

4) Periodic applications of Phosphorous, in a non-acid form, will ensure that there remains enough of this important compound in the soil to properly form a bond with the soil particles.

Further testing in the future, In Vitro Method for Determination of Lead and Arsenic Bioaccessibility, would substantiate the effectiveness of this process over time.

The success of this project is the result of the efforts and dedication of a number of people, including:

Mr. Randy Alvey	Alvey Laboratory, Inc.
Mr. Russ Batzel	City of St. Peters, Missouri
Ms. Sally Brown, Ph.D.	University of Washington
Ms Noemi Emeric	US EPA Region 5
Mr. Scott Fredericks	US EPA, Environmental Response Team
Mr. Kevin Turner	US EPA Region 5
Mr. Lue Walters	USDA NRCS

Project Budget:

	Expense	Amount
1)	Personnel	$15,000.00
2)	Materials (Compost freight)	$5,000.00
3)	Fencing	$500.00
4)	Custom Applications	$22,000.00
5)	Analysis/Testing	$2,750.00
6)	Waste Disposal	$350.00
7)	Administration	$4,400.00
	Total	$50,000.00

1) Includes personnel time to research the procedures, obtain permits, perform public outreach, maintain financials, develop reports, etc.
2) The Class "A" Biosolid compost was graciously donated by the City of St. Peters, Missouri. This figure represents the approximate freight costs associated with the 18 loads of materials delivered to the project site.
3) Fencing consisted of four foot high orange construction fence on steel "T" posts, at ten foot centers.
4) Custom applications includes permits, all labor to install (and uninstall) the fence and signs, disk the site, the addition of soil amendments (Calcium Carbonate, Phosphorous), removal of previously unidentified concrete slabs, re-disk the site, harrow to level, seed, fertilize and straw the site.
5) Includes all testing procedures previously listed within this document.
6) Removal of landscape waste from the site.

This figure represents an approximate amount that one would spend on a project of this nature in an inner-city environment. The removal of the abandoned concrete, as well as the need to remove all equipment from the site each night, for security reasons, greatly added to the costs on this particular site. The donation of the compost by the City of St. Peters had a very positive impact on the overall project.

How can I tell if my child may be poisoned?

Symptoms range from mild to severe. Anything from learning disabilities, hyperactivity, mild cold symptoms, mental retardation, not being able to sleep or loss of appetite can be some of the symptoms.

The best way to determine if your child may be poisoned is to have your doctor or local health department do a blood test.

Project Partners:
Southwestern Illinois RC&D, Inc.
US Environmental Protection Agency
USDA Natural Resources Conservation Service

Funding Provided by:
USDA Natural Resources Conservation Service

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East St. Louis Lead Remediation Project

Southwestern Illinois Resource Conservation & Development, Inc.

Southwestern Illinois RC&D, Inc. is a not-for-profit corporation, formed in 1989, to address regional natural resource concerns, to help find ways to solve regional issues and to assist in the development of the natural resources for the betterment of the community.

Questions about Lead Remediation

What causes lead to be found in community soils?

There are a number of causes of lead in community soils, including paint chips from lead-based paint, accumulated lead from auto-emissions and lead that has been deposited as a result of prior industrial practices. This project will focus on a former industrial site, Western Forge Works, which was located between 16th and 18th Streets, adjacent to St. Clair Avenue.

How do we know that lead is in the soil?

The Illinois Department of Public Health and US Environmental Protection Agency has done testing on this, and several other prior industrial sites, within East St. Louis. Their goal is to identify sites that are in need of remediation, and to then identify programs that will fund the clean-up efforts.

What can we do about lead-contaminated soil?

First and foremost we need to identify the contaminated sites and make certain that children, who are most susceptible to lead poisoning, are kept away from the site. The level of contamination and amount of funds that are available for treatment will dictate the course of action necessary to remediate the site. On this site, our organization will be performing a process called "in-place, inactivation".

How does the in-place inactivation process work?

For this project, we will be mixing a type of compost, called biosolids, with the soil. In addition, we will incorporate common nutrients, such as phosphorous and iron, to assist in the process. This blend of compost and nutrients will form a bond, between the lead that is present, and the soil. Once attached to the soil, the lead will not be able to leak onto other properties or into the groundwater. Turf grass is then planted on the site to further create a barrier between children and the soil.

If the lead is still in the soil, how does this process help?

By using this process, lead forms a very tight bond with the soil. Laboratory testing with rats shows that if lead-contaminated soil, that has gone through this process, is ingested by rats, it will pass through their bodies rather than being absorbed by the blood systems. The addition of the compost and nutrients also creates an excellent environment in which to grow plant material. Once established, turf grass will create a dense barrier between children and the lead.

How will the process be implemented, and how long will it take?

A construction fence will be placed around the site and we will ask for assistance from the community in monitoring the site during the course of the project. Tilling the site and adding the compost/nutrients will only take a few days. Once the materials have been added, grass seed and mulch will be applied to the site. It will take approximately one to two months for the seed to germinate and start to grow, depending on how much rain Mother Nature provides. The fence will be removed once the grass is well established.

For More Information, Call: (618) 566-4451

Solubility/Bioavailability Research Consortium

Standard Operating Procedure:

In Vitro Method for Determination of Lead and Arsenic Bioaccessibility

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1. Introduction

1.1 Synopsis

This SOP describes an *in vitro* laboratory procedure to determine a bioaccessibility value for lead or arsenic (i.e., the fraction that would be soluble in the gastrointestinal tract) for soils and solid waste materials. A recommended quality assurance program to be followed when performing this extraction procedure is also provided.

1.2 Purpose

An increasingly important property of materials/soils found at contaminated sites is the bioavailability of individual contaminants. Bioavailability is the fraction of a contaminant in a particular environmental matrix that is absorbed by an organism via a specific exposure route. Many animal studies have been conducted to experimentally determine the oral bioavailability of individual metals, particularly lead and arsenic. During the period 1989–1997, a juvenile swine model developed by EPA Region VIII was used to predict the relative bioavailability of lead and arsenic in approximately 20 soils/solid materials (Weis and LaVelle 1991; Weis et al. 1994; Casteel et al. 1997a,b). The bioavailability determined was relative to that of a soluble salt (i.e., lead acetate trihydrate or sodium arsenate). The tested materials had a wide range of mineralogy, and produced a range of lead and arsenic bioavailability values. In addition to the swine studies, other animal models (e.g., rats and monkeys) have been used to measure the bioavailability of lead and arsenic from soil.

Several researchers have developed *in vitro* tests to measure the fraction of a chemical solubilized from a soil sample under simulated gastrointestinal conditions. This measurement is referred to as “bioaccessibility” (Ruby et al. 1993). Bioaccessibility is thought to be an important determinant of bioavailability, and several groups have sought to compare bioaccessibility determined in the laboratory to bioavailability determined in

animal studies (Imber 1993; Ruby et al. 1996; Medlin 1997; Rodriguez et al. 1999). The *in vitro* tests consist of an aqueous fluid, into which soils containing lead and arsenic are introduced. The solution then solubilizes the soil under simulated gastric conditions. Once this procedure is complete, the solution is analyzed for lead and/or arsenic concentration. The mass of lead and/or arsenic found in the aqueous phase, as defined by filtration at the 0.45-μm pore size, is compared to the mass introduced into the test. The fraction liberated into the aqueous phase is defined as the bioaccessible fraction of lead or arsenic in that soil. To date, for lead-bearing soils tested in the EPA swine studies, this *in vitro* method has correlated well with relative bioavailability values.

2. Procedure

2.1 Sample Preparation

All soil/material samples should be prepared for testing by oven drying (<40 °C) and sieving to <250 μm. The <250-μm size fraction is used because this particle size is representative of that which adheres to children's hands. Subsamples for testing in this procedure should be obtained using a sample splitter.

2.2 Apparatus and Materials

2.2.1 Equipment

The main piece of equipment required for this procedure consists of a Toxicity Characteristic Leaching Procedure (TCLP) extractor motor that has been modified to drive a flywheel. This flywheel in turn drives a Plexiglass block situated inside a temperature-controlled water bath. The Plexiglass block contains ten 5-cm holes with stainless steel screw clamps, each of which is designed to hold a 125-mL wide-mouth high-density polyethylene (HDPE) bottle (see Figure 1). The water bath must be filled such that the extraction bottles are immersed. Temperature in the water bath is maintained at 37±2 °C using an immersion circulator heater (for example, Fisher Scientific Model 730). Additional equipment for this method includes typical laboratory supplies and reagents, as described in the following sections.

The 125-mL HDPE bottles must have an air-tight screw-cap seal (for example, Fisher Scientific 125-mL wide-mouth HDPE Cat. No. 02-893-5C), and care must be taken to ensure that the bottles do not leak during the extraction procedure.

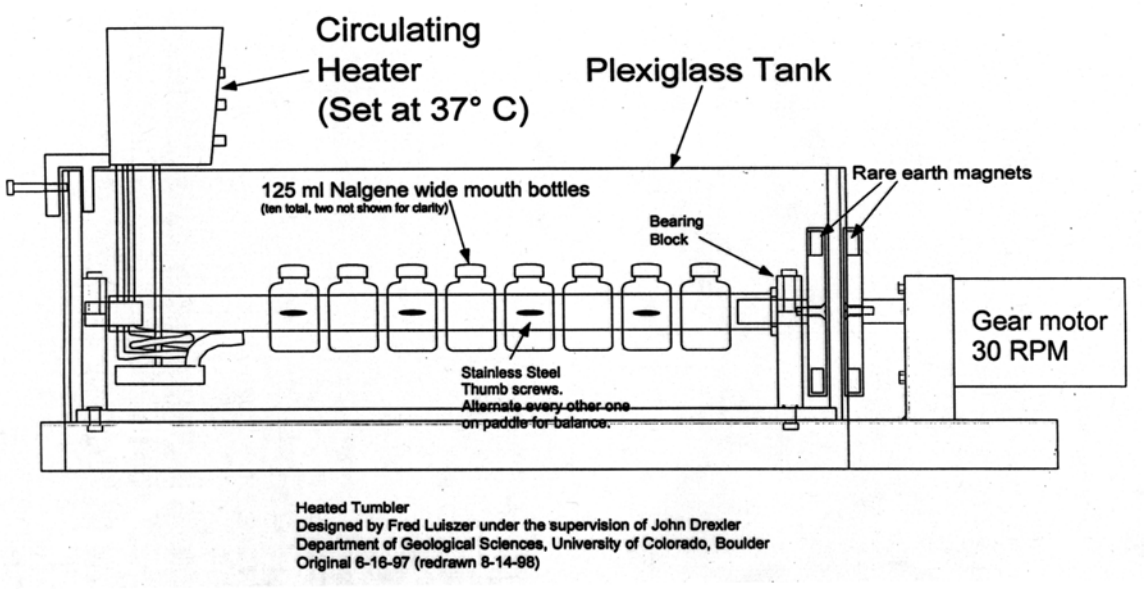

Figure 1. Extraction device for performing the SBRC *in vitro* extraction

2.2.2 Standards and Reagents

The leaching procedure for this method uses a buffered extraction fluid at a pH of 1.5. The extraction fluid is prepared as described below.

The extraction fluid should be prepared using ASTM Type II deionized (DI) water. To 1.9 L of DI water, add 60.06 g glycine (free base, Sigma Ultra or equivalent). Place the mixture in a water bath at 37 °C until the extraction fluid reaches 37 °C. Standardize the pH meter using temperature compensation at 37 °C or buffers maintained at 37 °C in the water bath. Add concentrated hydrochloric acid (12.1 N, Trace Metal grade) until the solution pH reaches a value of 1.50 ±0.05 (approximately 120 mL). Bring the solution to a final volume of 2 L (0.4 M glycine).

Cleanliness of all reagents and equipment used to prepare and/or store the extraction fluid is essential. All glassware and equipment used to prepare standards and reagents must be properly cleaned, acid washed, and finally, rinsed with DI water prior to use. All reagents must be free of lead and arsenic, and the final fluid should be tested to confirm that lead and arsenic concentrations are less than 25 and 5 μg/L, respectively.

2.3 Leaching Procedure

Measure 100 ±0.5 mL of the extraction fluid, using a graduated cylinder, and transfer to a 125-mL wide-mouth HDPE bottle. Add 1.00 ±0.05 g of test substrate (<250 μm) to the bottle, ensuring that static electricity does not cause soil particles to adhere to the lip or outside threads of the bottle. If necessary, use an antistatic brush to eliminate static electricity prior to adding the soil. Record the volume of solution and mass of soil added to the bottle on the extraction test checklist (see Attachment A for example checklists). Hand-tighten each bottle top, and shake/invert to ensure that no leakage occurs, and that no soil is caked on the bottom of the bottle.

Place the bottle into the modified TCLP extractor, making sure each bottle is secure and the lid(s) are tightly fastened. Fill the extractor with 125-mL bottles containing test materials or Quality Control samples.

The temperature of the water bath must be 37±2 °C. Record the temperature of the water bath at the beginning and end of each extraction batch on the appropriate extraction test checklist sheet (see Attachment A).

Rotate the extractor end over end at 30±2 rpm for 1 hour. Record start time of rotation.

When extraction (rotation) is complete, immediately remove bottles, wipe them dry, and place them upright on the bench top.

Draw extract directly from reaction vessel into a disposable 20-cc syringe with a Luer-Lok attachment. Attach a 0.45-μm cellulose acetate disk filter (25 mm diameter) to the syringe, and filter the extract into a clean 15-mL polypropylene centrifuge tube or other appropriate sample vial for analysis. Store filtered sample(s) in a refrigerator at 4 °C until they are analyzed.

Record the time that the extract is filtered (i.e., extraction is stopped). If the total elapsed time is greater than 1 hour 30 minutes, the test must be repeated.

Measure and record the pH of fluid remaining in the extraction bottle. If the fluid pH is not within ±0.5 pH units of the starting pH, the test must be discarded and the sample reanalyzed as follows.

If the pH has dropped by 0.5 or more pH units, the test will be re-run in an identical fashion. If the second test also results in a decrease in pH of greater than 0.5 s.u., the pH will be recorded, and the extract filtered for analysis. If the pH has increased by 0.5 or more units, the test must be repeated, but the extractor must be stopped at specific intervals and the pH manually adjusted down to pH 1.5 with dropwise addition of HCl (adjustments at 5, 10, 15, and 30 minutes into the extraction, and upon final removal from the water bath [60 minutes]). Samples with rising pH values must be run in a separate extraction, and must not be combined with samples being extracted by the standard method (continuous extraction).

Extracts are to be analyzed for lead and arsenic concentration using analytical procedures taken from the U.S. EPA publication, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846*. (current revisions). Inductively coupled plasma (ICP) analysis, method 6010B (December 1996 revision) will be the method of choice. This method should be adequate for determination of lead concentrations in sample extracts, at a project-required detection limit (PRDL) of 100 μg/L. The PRDL of 20 μg/L for arsenic may be too low for ICP analysis for some samples. For extracts that have arsenic concentrations less than five times the PRDL (e.g., <100 μg/L arsenic), analysis by ICP-hydride generation (method 7061A, July 1992 revision) or ICP-MS (method 6020, September 1994 revision) will be required.

2.4 Calculation of the Bioaccessibility Value

A split of each solid material (<250 μm) that has been subjected to this extraction procedure should be analyzed for total lead and/or arsenic concentration using analytical procedures taken from the U.S. EPA publication, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. SW-846*. (current revisions). The solid material should be acid digested according to method 3050A (July 1992 revision) or method 3051 (microwave-assisted digestion, September 1994 revision), and the digestate analyzed for lead and/or arsenic concentration by ICP analysis (method 6010B). For samples that have arsenic concentrations below ICP detection limits, analysis by ICP-hydride generation (method 7061A, July 1992 revision) or ICP-MS (method 6020, September 1994 revision) will be required.

The bioaccessibility of lead or arsenic is calculated in the following manner:

$$Bioaccessibility\ value = \frac{(concentration\ in\ in\ vitro\ extract,\ mg/L)\ (0.1L)}{(concentration\ in\ solid,\ mg/kg)\ (0.001\ kg)} \times 100$$

2.5 Chain-of-Custody/Good Laboratory Practices

All laboratories that use this SOP should receive test materials with chain-of-custody documentation. When materials are received, each laboratory will maintain and record custody of samples at all times. All laboratories that perform this procedure should follow good laboratory practices as defined in 40 CFR Part 792 to the extent practical and possible.

2.6 Data Handling and Verification

All sample and fluid preparation calculations and operations should be recorded in bound and numbered laboratory notebooks, and on extraction test checklist sheets. Each page must be dated and initialed by the person who performs any operations. Extraction and

filtration times must be recorded, along with pH measurements, adjustments, and buffer preparation. Copies of the extraction test checklist sheets should accompany the data package.

3. Quality Control Procedures

3.1 Elements of Quality Assurance and Quality Control (QA/QC)

A standard method for the *in vitro* extraction of soils/solid materials, and the calculation of an associated bioaccessibility value, are specified above. Associated QC procedures to ensure production of high-quality data are as follows (see Table 1 for summary of QC procedures, frequency, and control limits):

- Reagent blank—Extraction fluid analyzed once per batch.
- Bottle blank—Extraction fluid only run through the complete extraction procedure at a frequency of no less than 1 per 20 samples or one per extraction batch, whichever is more frequent.
- Blank spikes—Extraction fluid spiked at 10 mg/L lead and/or 1 mg/L arsenic and run through the extraction procedure at a frequency of no less than every 20 samples or one per extraction batch, whichever is more frequent. Blank spikes should be prepared using traceable 1,000-mg/L lead and arsenic standards in 2 percent nitric acid.
- Duplicate—duplicate extractions are required at a frequency of 1 for every 10 samples. At least one duplicate must be performed on each day that extractions are conducted.
- Standard Reference Material (SRM)—National Institute of Standards and Technology (NIST) material 2711 (Montana Soil) should be used as a laboratory control sample (LCS).

Control limits for these QC samples are delineated in Table 1, and in the following discussion.

Table 1. Summary of QC samples, frequency of analysis, and control limits

QC Sample	Minimum Frequency of Analysis	Control Limits
Reagent Blank	Once per batch (min. 5%)	<25 μg/L lead <5 μg/L arsenic
Bottle Blank	Once per batch (min. 5%)	<50 μg/L lead <10 μg/L arsenic
Blank Spike	Once per batch (min. 5%)	85–115% recovery
Duplicate	10%	±20% RPD
SRM (NIST 2711)	2%	9.22 ±1.50 mg/L Pb 0.59 ±0.09 mg/L As

3.2 QA/QC Procedures

Specific laboratory procedures and QC steps are described in the analytical methods cited in Section 2.3, and should be followed when using this SOP.

3.2.1 Laboratory Control Sample (LCS)

The NIST SRM 2711 should be used as a laboratory control sample for the *in vitro* extraction procedure. Analysis of 18 blind splits of NIST SRM 2711 (105 mg/kg arsenic and 1,162 mg/kg lead) in four independent laboratories resulted in arithmetic means ± standard deviations of 9.22 ±1.50 mg/L lead and 0.59 ±0.09 mg/L arsenic. This SRM is available from the National Institute of Standards and Technology, Standard Reference Materials Program, Room 204, Building 202, Gaithersburg, Maryland 20899 (301/975-6776).

3.2.2 Reagent Blanks/Bottle Blanks/Blank Spikes

Reagent blanks must not contain more than 5 μg/L arsenic or 25 μg/L lead. Bottle blanks must not contain arsenic and/or lead concentrations greater than 10 and 50 μg/L, respectively. If either the reagent blank or a bottle blank exceeds these values, contamination of reagents, water, or equipment should be suspected. In this case, the laboratory must investigate possible sources of contamination and mitigate the problem

before continuing with sample analysis. Blank spikes should be within 15% of their true value. If recovery of any blank spike is outside this range, possible errors in preparation, contamination, or instrument problems should be suspected. In the case of a blank spike outside specified limits, the problems must be investigated and corrected before continuing sample analysis.

4. References

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Extraction Fluid Preparation

Date of Extraction Fluid Preparation:_____ Prepared by:_____

Extraction Fluid Lot #:_____

Component	Lot Number	Fluid Preparation 1L	Fluid Preparation 2L	Acceptance Range	Actual Quantity	Comments
Deionized Water		0.95 L (approx.)	1.9 L (approx.)	---		
Glycine		30.03±0.05 g	60.06±0.05g	---		
HCl [a]		60 mL (approx.)	120 mL (approx.)	---		
Final Volume	---	1 L (Class A, vol.)	2 L (Class A, vol.)	---		
Extraction Fluid pH value (@ 37°C)	---	1.50±0.05	1.50±0.05	1.45–1.55		

[a] Concentrated hydrochloric acid (12.1 N)

Required Parameters:

Volume of extraction fluid (V) = 100 ±0.5 mL

Mass of test substrate (M) = 1.00 ±0.05 g

Temperature of water bath = 37 ±2 °C

Extraction time = 60 ±5 min

Extractor rotation speed = 30 ±2 rpm

Maximum elapsed time from extraction to filtration = 90 minutes

Maximum pH difference from start to finish (ΔpH)= 0.5 pH units

Spike solution concentrations: As = 1 mg/L; Pb = 10 mg/L

Date of Extraction:_____

Extraction Fluid Lot #:_____

Extracted by:_____

As Spike Solution Lot #:_____

Pb Spike Solution Lot #:_____

Extraction Log:

Sample ID	Sample Preparation		Extraction								Filtration	
	V (mL)	M (g)	Start Time[a]	End Time[a]	Elapsed Time (min)	Start pH	End pH	ΔpH	Start Temp (°C)	End Temp (°C)	Time[a]	Time Elasped from extraction (min)
Acceptance Range	(95.5–100.5)	(0.95–1.05)	---	---	(55–65 min)	---	---	(Max = 0.5)	(35–39)	(35–39)		(Max = 90 min)
Bottle Blank												
Matrix spike												

[a] 24-hour time scale

Analytical Procedures

QC Requirements:

QC Sample	Minimum Analysis Frequency	Control Limits	Corrective Action[a]
Reagent blank	once per batch (min. 5%)	< 25 µg/L Pb <5 µg/L As	Investigate possible sources of target analytes. Mitigate contamination problem before continuing analysis.
Bottle blank	once per batch (min. 5%)	< 50 µg/L Pb <10 µg/L As	Investigate possible sources of target analytes. Mitigate contamination problem before continuing analysis.
Blank spike	once per batch (min. 5%)	85–115%	Re-extract and reanalyze sample batch
Duplicate	10% (min. once/day)	±20% RPD	Re-homongenize, re-extract and reanalyze

RPD – Relative percent difference
a – Action required if control limits are not met